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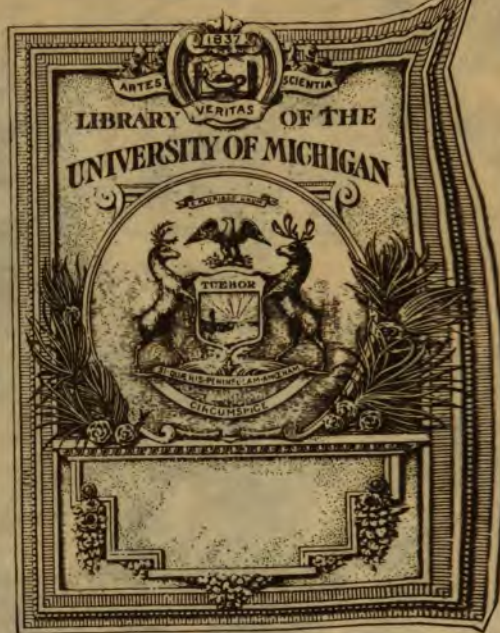
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NOTES ON ASSAYING





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# NOTES ON ASSAYING.

BY

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PIERRE DE P. RICKETTS.

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## PREFACE.

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RECENT progress in the art of assaying as it was defined years ago, and in those rapid quantitative determinations now generally termed assays, has made necessary the present manual of assaying, which is designed to replace "Notes on Assaying and Assay Schemes," published in 1876 and revised in 1879. The general arrangement is the same, and it is hoped that the present work will fill as well the wants of both the practical assayer and the student.

Many new and rapid methods are included, taken from various journals, to which the references are given, but in no case has any method been described which has not been found successful when tested in the Assay Laboratory of the School of Mines, Columbia University.

Some familiarity with chemical operations and apparatus is presupposed, so that the ordinary analytical manipulations are but briefly mentioned, and only the special details necessary to the success of the method given in full.

We have not adopted the new chemical spelling, as it is not as yet in general use among practical men.

This work embodies the system of assaying practiced in Columbia University, as organized and developed by Prof. C. F. Chandler, and by G. M. Miller, E. C. H. Day, F. Prime,

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Jr., T. M. Blossom, and the authors of these notes, who have successively had charge of the Assay Laboratory.

We take pleasure in acknowledging our indebtedness to Prof. A. J. Moses for the use of his blowpipe tables, to Dr. H. T. Vulté for the qualitative schemes arranged specially for this book, to Dr. J. H. Banks for revision of the laboratory tests on ores, to Dr. P. C. McIlhiney for the bromine extraction test, and to Mr. J. A. Mathews, Assistant in Assaying, for aid in testing the charges and methods given.

PIERRE DE PEYSTER RICKETTS,  
EDMUND H. MILLER.

ASSAY LABORATORY, COLUMBIA UNIVERSITY, November, 1896.

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## **PART I.**

**INTRODUCTION, APPARATUS, REAGENTS AND  
CHEMICALS, OPERATIONS.**





## INTRODUCTION.

---

ASSAYING has for its object the determination of the elements in their various compounds, such as ores, alloys, and furnace products.

The rules are empirical, and a knowledge of chemistry is not absolutely necessary, although the assayer will find that a familiarity with chemical laws and reactions will greatly facilitate his work. The following is a list of the elementary bodies, with the exception of one or two recently discovered, and their atomic weights and symbols. An element is a body which chemical research has failed to reduce to a more simple form, or separate into constituent parts. The symbol of an element is generally the first letter or letters of its Latin name; and its atomic weight is the smallest amount of that element which will enter into combination with other elements; the first column of figures gives the atomic mass referred to hydrogen as unity, the second to oxygen as sixteen. Both sets of values are given, as recent determinations have shown that the ratio of oxygen to hydrogen is not exactly 16 : 1.

## ATOMIC MASSES.

| Name.           | H = 1. | O = 16. | Name.           | H = 1.  | O = 16. |
|-----------------|--------|---------|-----------------|---------|---------|
| Aluminium.....  | 26.91  | 27.11   | Molybdenum..... | 95.26   | 95.98   |
| Antimony.....   | 119.52 | 120.43  | Neodymium.....  | 139.4   | 140.5   |
| Argon.....      | ?      | ?       | Nickel.....     | 58.24   | 58.69   |
| Arsenic.....    | 74.52  | 75.09   | Nitrogen.....   | 13.94   | 14.04   |
| Barium.....     | 136.40 | 137.43  | Osmium.....     | 189.55  | 190.99  |
| Bismuth.....    | 206.54 | 208.11  | Oxygen.....     | 15.879  | 16.00   |
| Boron.....      | 10.86  | 10.95   | Palladium.....  | 105.56  | 106.86  |
| Bromine.....    | 79.84  | 79.95   | Phosphorus..... | 30.79   | 31.02   |
| Cadmium.....    | 111.08 | 111.93  | Platinum.....   | 193.41  | 194.89  |
| Cæsium.....     | 131.89 | 132.89  | Potassium.....  | 38.82   | 39.11   |
| Calcium.....    | 39.78  | 40.08   | Præodymium..... | 142.4   | 143.5   |
| Carbon.....     | 11.92  | 12.01   | Rhodium.....    | 102.23  | 103.01  |
| Cerium.....     | 139.1  | 140.2   | Rubidium.....   | 84.78   | 85.43   |
| Chlorine.....   | 35.18  | 35.45   | Ruthenium.....  | 100.91  | 101.68  |
| Chromium.....   | 51.74  | 52.14   | Samarium.....   | 148.9   | 150.0   |
| Cobalt.....     | 58.49  | 58.93   | Scandium.....   | 43.7    | 44.0    |
| Columbium.....  | 93.3   | 94.0    | Selenium.....   | 78.4    | 79.0    |
| Copper.....     | 63.12  | 63.60   | Silicon.....    | 28.18   | 28.40   |
| Erbium.....     | 165.0  | 166.3   | Silver.....     | 107.11  | 107.92  |
| Fluorine.....   | 18.89  | 19.03   | Sodium.....     | 22.88   | 23.05   |
| Gadolinium..... | 154.9  | 156.1   | Strontium.....  | 86.95   | 87.61   |
| Gallium.....    | 68.5   | 69.0    | Sulphur.....    | 31.83   | 32.07   |
| Germanium.....  | 71.75  | 72.3    | Tantalum.....   | 181.2   | 182.6   |
| Glucinum.....   | 9.01   | 9.08    | Tellurium.....  | 126.1 ? | 127.0 ? |
| Gold.....       | 195.74 | 197.24  | Terbium.....    | 158.8   | 160.0   |
| Helium.....     | ?      | ?       | Thallium.....   | 202.60  | 204.15  |
| Hydrogen.....   | 1.0    | 1.008   | Thorium.....    | 230.87  | 232.63  |
| Indium.....     | 112.8  | 113.7   | Thulium.....    | 169.4   | 170.7   |
| Iodine.....     | 125.89 | 126.85  | Tin.....        | 118.15  | 119.05  |
| Iridium.....    | 191.66 | 193.12  | Titanium.....   | 47.79   | 48.15   |
| Iron.....       | 55.60  | 56.02   | Tungsten.....   | 183.44  | 184.84  |
| Lanthanum.....  | 137.6  | 138.6   | Uranium.....    | 237.77  | 239.59  |
| Lead.....       | 205.36 | 206.92  | Vanadium.....   | 50.99   | 51.38   |
| Lithium.....    | 6.97   | 7.03    | Ytterbium.....  | 171.7   | 173.0   |
| Magnesium.....  | 24.11  | 24.29   | Yttrium.....    | 88.28   | 88.95   |
| Manganese.....  | 54.57  | 54.99   | Zinc.....       | 64.91   | 65.41   |
| Mercury.....    | 198.5  | 200.0   | Zirconium.....  | 89.9    | 90.6    |

The above table of the atomic weights of the elements was printed for the use of the students of the School of Mines, and contains the latest values as reported by the committee on atomic weights to the American Chemical Society, and published in the Journal Am. Chem. Soc., Mch., 1896.

The various methods for the determination of the metals in their compounds may be classed under two heads:

- 1st. "Dry way," or assaying proper.
- 2d. "Wet way," or analysis.

The first includes all determinations by the direct action of heat, the various operations being performed in furnaces.

The second head embraces the estimation and separation of the elements by the action of solvents aided or unaided by heat, the use of furnaces not being essential.

There are many cases, of course, where the first class merges into the second, and *vice versa*.

It was originally intended to give in the following pages only a few concise methods for the estimation of the metals in ores and other compounds by fire assay; but, as in many cases the dry methods have been replaced by volumetric methods which are both rapid and accurate, these are given in Part III. Further, as some metals are associated with others which are either of value or detriment, and whose determination is often necessary, a few schemes for the treatment of such compounds in the wet way have been added.

The various operations which may take place in making an assay proper, are—

- 1st. Preliminary testing of the ore.
- 2d. Preparation of the ore, sampling, pulverizing, etc.
- 3d. Weighing out the ore and reagents.
- 4th. Drying, calcination, and roasting.
- 5th. Reduction and fusion.

- 6th. Distillation and sublimation.
- 7th. Scorification and cupellation.
- 8th. Inquartation and parting, including solution.
- 9th. Weighing beads and bullion.
- 10th. Tabulating results and reporting.

All of the above will be described further on ; but as some of the operations require great care in their performance, a few rules and hints for the guidance of the beginner may not be out of place.

1st. Sample well and carefully, for without a fair sample the assay will be useless.

2d. Weigh carefully, and adjust the balance before and after weighing.

3d. Always weigh an ore before drying, calcining, or roasting, and always roast thoroughly.

4th. Never fill a crucible or scorifier more than three-quarters full, and when a crucible is removed from the fire, tap it gently on the floor to settle the metal, unless otherwise directed, and keep the crucible covered.

5th. To break a crucible, hit with a medium-sized riveting hammer near the centre, so as to break off the top at one blow. Then lay the bottom on the anvil, and crack it through to get the button out whole. Never break until perfectly cold.

To break a scorifier, lay it bottom up on the anvil ; encircle with the hand, and then strike the bottom. The button will generally come out free from slag.

6th. Never take a scorification or cupellation from the furnace to finish at a future time, but complete the operation at once. When buttons are to be scorified or cu-

pelled, be sure that they are free from moisture, and heat gradually.

7th. Be certain that all reagents used in an assay are dry and pure, especially when testing for the precious metals.

8th. In reporting results, recollect that a fire assay does not always give the exact amount of metal contained, but often shows only what the yield of an ore would be in smelting, and that the assay of a small piece of ore cannot represent the average value of the bed or vein from which it may have been taken, and word your report accordingly.

9th. Always observe the color and character of the slag produced in an assay, as the nature of the ore treated may often be determined in this way.

10th. Never accept the results of an assay where the fusion has been incomplete, the button formed being small or brittle ; where the scorification slag is lumpy or masses of ore are found adhering to the sides of the scorifier, or when the button freezes or sprouts in cupellation.

## MANIPULATION, FORMULÆ, AND CALCULATION.

The various operations of weighing, mixing, charging, etc., will be described under their appropriate heads ; and it only remains to give a few hints on operations necessary in the performance of assays in the wet way.

**PRECIPITATION.**—This operation is the sudden conversion of a dissolved body into the solid state, either by a modification of the solvent, or decomposition with the formation of a new compound.

The separation of a precipitate is generally aided by the action of heat and agitation.

Porcelain and glass vessels are the best.

In adding the necessary reagent pour in carefully until the precipitate ceases to form ; unless otherwise directed, a large excess of the precipitant should be avoided.

**FILTRATION.**—This operation has for its object the separation of the solid particles suspended in a fluid, from the same. Various substances might be used as a filter, but the best is unsized paper, which is prepared for the purpose by cutting a circular form and then folding it into halves and quarters, so that it will just fit into a funnel and not project above the rim. For quantitative work, the prepared Swedish filter paper will be found the best ; it should be cut into circular pieces as described, which should be of a size to suit the funnels ; and the ash left by

burning one of the same carefully determined. Filter papers practically free from mineral matter are also used. As a rule, it will be found best to moisten the paper with water before filtering, and to pour on first the fluid portion of the mixture to be filtered.

**DÉCANTATION.**—This is simply a substitute for filtration, the clear liquor being poured off from the precipitate. To effect this the liquid is permitted to run down a glass rod held against the spout of the vessel, which should be inclined gently, so as not to disturb the precipitate.

**WASHING.**—This is best effected by using a glass flask, fitted with a cork, in which are inserted two glass tubes, one reaching to the bottom of the flask, and bent to any desired angle on the outside, the end being drawn to a point. The second tube reaches to just below the cork, and is also bent on the outside, but not drawn to a point; by blowing in this tube the water is expelled through the first. Warm water will be found the most effective. The completeness of the washing may be tested by evaporating a small portion of the filtrate on platinum foil, and noting the residue, or by suitable qualitative tests.

**EVAPORATION.**—Porcelain dishes are the best for this operation; but if the solution is to be evaporated to dryness it should be conducted over a water bath. A sand bath may be used, but care should be taken to prevent loss by spattering.

**IGNITION.**—The washed precipitate, after being dried, is ignited to completely expel all moisture, or convert it into a constant or weighable substance. This is best conducted by transferring to a weighed platinum crucible, and burning the filter paper over it, either on the inverted cover, or by wrapping it in a coil of platinum wire and



holding it over the crucible. The ash should be heated until white, or nearly so. The whole operation must be conducted over a piece of glazed paper until the filter paper is burnt, after which the crucible and contents should be heated over a burner or lamp; gently at first. The oxidation of organic or carbonaceous matter can often be hastened by adding a drop of nitric acid and then carefully heating. After ignition the crucible and contents should be cooled in a desiccator, to avoid absorption of moisture from the air.

**FORMULÆ AND CALCULATION.**—The general methods of calculation will be given under the various assays, but it will be well to bear in mind the following:

1st. The equivalent of the compound found is to the equivalent of its constituent which is sought as the weight of the compound is to the weight of the constituent.

2d. The weight of the substance taken for assay is to the weight of the constituent sought as one hundred is to the per cent. of the same.

The equivalents (atomic weights) will be found in the table on page 4. The equivalent of a compound is equal to the sum of the equivalents of the constituents of the same. Thus,  $\text{H}_2\text{SO}_4$  (sulphuric acid) is equal to  $2.016 + 32.07 + 64 = 98.086$ . The equivalent of hydrogen (H) being 1.008, sulphur (S) = 32.07, oxygen (O) = 16. Two parts of hydrogen being 2.016, four parts of oxygen = 64.

Suppose in a coal analysis the percentage of sulphur is required, and that 1.5 grams of coal gave 0.2137 grams of barium sulphate. First, make the proportion:

$$\begin{array}{ccccccc} 0.2137 & : & x & :: & 233.5 & : & 32.07. \\ \text{wt. BaSO}_4 & & \text{wt S} & & \text{molecular wt. BaSO}_4 & & \text{atomic wt. S} \end{array}$$

Then  $x$  (wt. S in 1.5 grams coal) :  $y$  (the percentage of sulphur) :: 1.5 : 100, or  $\frac{x}{1.5} 100 = \% \text{ sulphur}$ .

## BALANCES AND WEIGHTS.

Four balances will be found useful in an assay laboratory.

*a.*—A rough scale for weighing large samples of ores, metals, fluxes in bulk, etc. An ordinary grocer's scale will do for this purpose.

*b.*—A balance for weighing out ore for assay, and the buttons of the base metals. (Fig. 1.) This balance should



FIG. 1.

take ten ounces in each pan, turn with one-twentieth of a grain, and be provided with movable pans, level, and set-

screws for adjusting. It is generally placed on a box, furnished with a drawer for weights.

*c.*—An analytical balance, protected by a glass case, having agate bearings, sensitive to ( $\frac{1}{20}$ ) one-twentieth of a milligram with a load of a hundred grams (See Fig. 2).

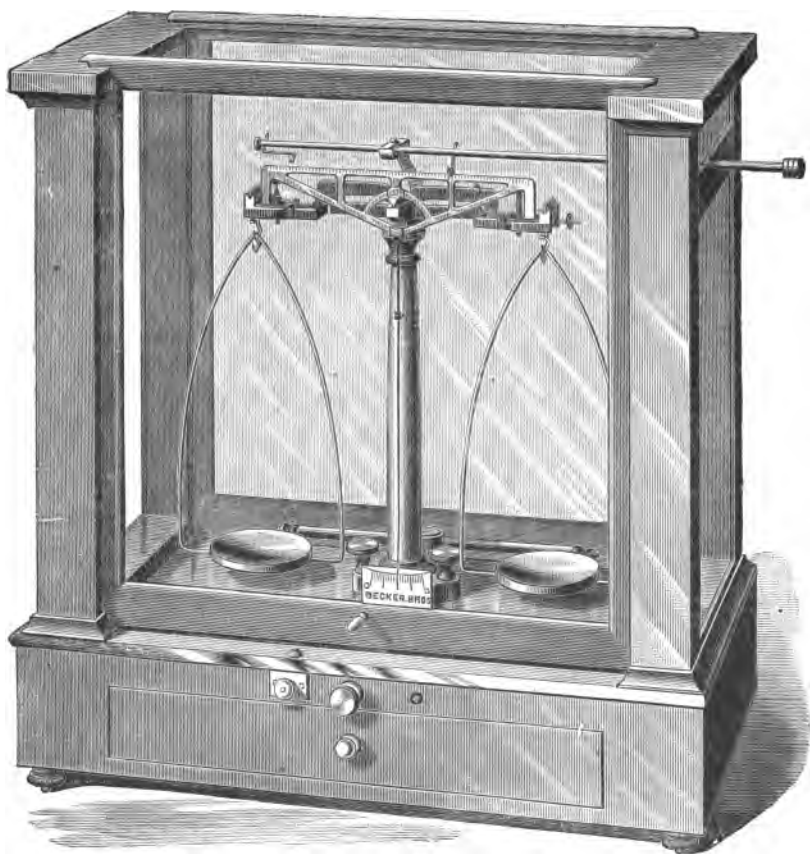


FIG. 2.—MODERN ANALYTICAL SHORT BEAM BALANCE.

The short beam balance is to be preferred.

*d.*—A bullion balance, similar to the analytical balance in construction but smaller and more sensitive. This bal-

ance is the most important, as well as the most expensive piece of apparatus in the assayer's outfit, and must be used with great care.

We have found a balance made by Becker with a short aluminium beam graduated to fiftieths of a milligram on both sides with agate bearings, and platinum pans supported on agate, most satisfactory. This balance is mounted on a thick glass plate and is capable of weighing accurately to one hundredth of a milligram, see Fig. 3.

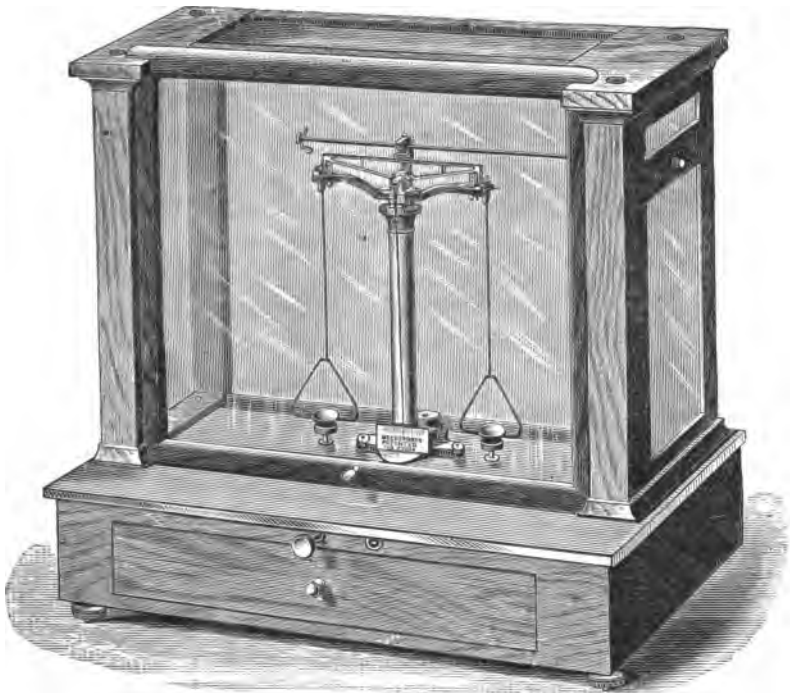


FIG. 3.—BULLION BALANCE.

To adjust this balance remove all dust with a camels-hair brush, level exactly by turning screws underneath the balance, then turn the small screw on the right end of

the beam, very carefully steadying the hand against the balance case, until the indicator swings the same number of divisions on either side.

THE WEIGHTS employed by the assayer may be classified as :

*a.*—Avoirdupois for ores, base metals, and fluxes.

*b.*—Troy for gold, silver, and platinum.

*c.*—The French system based upon the gram as a unit. These weights can be used for weighing ores, fluxes, buttons, and precipitates, and will always be found most convenient, as they are on the scale of ten.

*d.*—The assay weights, which is a system made up from a comparison of the three foregoing, will be found extremely simple and useful, saving a vast amount of calculation and labor (see table, page 253).

The unit of the system is the assay ton = 29.166 grams. Its derivation will be seen at a glance.

One lb. Avoirdupois = 7,000 Troy grains.

2,000 lbs. = one ton.

$2,000 \times 7,000 = 14,000,000$  Troy grains, in one ton Avoirdupois.

480 Troy grains = 1 oz. Troy.

$14,000,000 \div 480 = 29,166 \frac{2}{3}$  Troy ozs. in 2,000 lbs. Avoirdupois.

There are 29,166 milligrams in one assay ton (A. T.); hence—

2,000 lbs. is to 1 A. T., as 1 oz. Troy is to 1 milligram.

EXAMPLE.—Weigh an A. T. of ore, and if on assay it yields 1 milligram of gold or silver, the result reads one Troy oz. in 2,000 lbs. Avoirdupois, without further calculation.

Besides Avoirdupois and Troy weights the assayer will find the following necessary—

1st. Gram weights from 50 grams to 10 milligrams (milligrams and fractions of a milligram are estimated by the rider) for weighing ore, etc., to be assayed by wet methods, also for weighing buttons of base metal.

2nd. Assay ton weights .4 A. T. to  $\frac{1}{10}$  A. T. for gold and silver ores.

3rd. A most accurate set of weights from 1 gram. to 1 milligram, for the bullion balance.

### FURNACES AND FUELS.

1st. FURNACE FOR CALCINING OR ROASTING. — Fig. 4. represents two sections.

The fireplace is made shallow; and, as a high temperature is not required, it may be made of red brick, or only lined with fire-brick, and the body of the furnace bound with strap-iron.

It should also have a cast-iron top-plate.

The grate-bars may be in one piece or separate, and draw out. The ash-pit should be provided with a door, which may be closed or opened in order to regulate the draft.

A hood of sheet-iron will

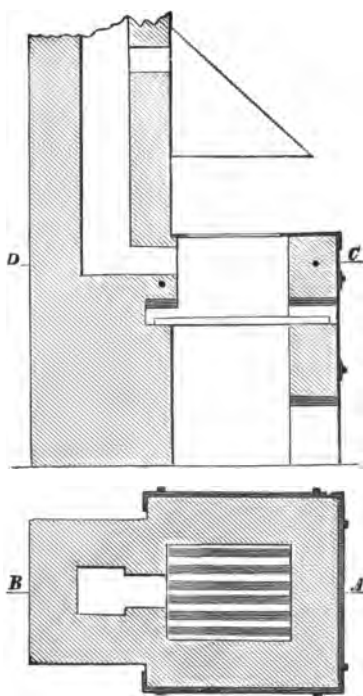


FIG. 4.

Scale  $\frac{1}{4}$  inch to the foot.

also be found necessary in many cases, as the fumes given off in roasting are often injurious. It is an excellent plan to have the hood of galvanized iron to prevent rusting.

The chimney may be of brick, iron, or clay.

2d. FURNACES FOR FUSION OR MELTING (Figs. 5 and 6).—These furnaces should be deeper than the preceding

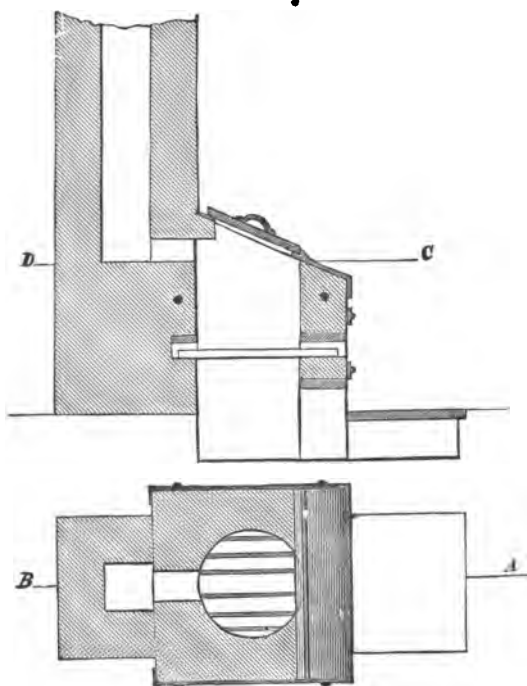


FIG. 5.

Scale  $\frac{1}{4}$ -inch to the foot.

one, and like it, may be built of red brick ; but it is better to line them with fire-brick.

For heavy work the furnaces should be low, to facilitate the lifting in and out of crucibles. Sometimes a crane is added for this purpose.

The chimney ought to be of brick, and the higher it is, the stronger will be the draft. This may be regulated by a damper as well as by the ash-pit door.

The tops should be of cast-iron, and the cover lift or slide easily. An iron shelf can be placed in front to hold ingot moulds when metals are to be poured, as shown in Fig. 6, which shows in section the furnace used for deposit melting in the United States mints.

3d. MUFFLE FURNACES FOR SCORIFICATION AND CUPELLATION.—Fig. 7 shows section of a portable cupel furnace.

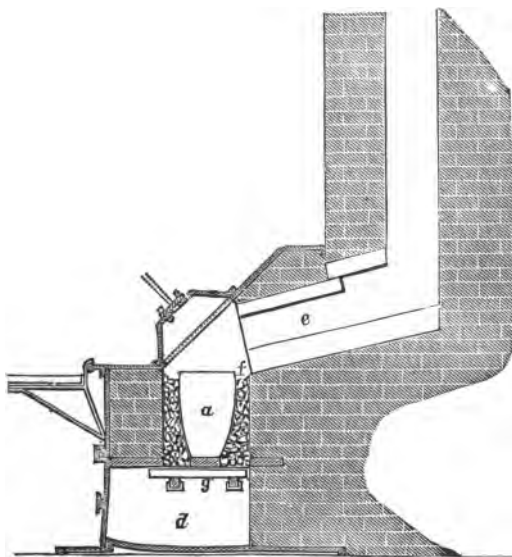


FIG. 6.

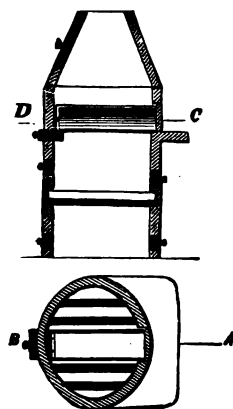
Scale  $\frac{1}{4}$ -inch to the foot.

FIG. 7.

Scale  $\frac{3}{8}$ -inch to the foot.

The same furnace may be used for both operations, but generally it will be found convenient to have a larger muffle for scorification and a higher heat.



The muffles are made of refractory clay, and in one piece; and should be thoroughly dried before using.

The draft of the furnace ought to be sufficient to carry off lead fumes, which are injurious. The construction of the furnace will vary with the fuel used and work to be done.

Fig. 8 is an assay furnace for the use of soft coal. This furnace has the following advantages: 1st. Economy of fuel. A furnace containing 8x14-inch muffles may be run for eight hours with not more than 100 lbs. of coal. Any tree-burning coal may be used (the Cañon City coal of Colorado—a lignite—gives very excellent results at Pueblo). 2d. Economy of construction. Very few fire-brick and other material are required, and the furnace may be built, exclusive of stack, for from \$25 to \$50. 3d. Saving in muffles. The muffle not being in contact with the fuel, and subjected only to the action of the flame and gases, does not become covered with slag, but always remains clean and is easily heated. When two muffles are employed, the upper one is sufficiently hot for cupellation, but not for scorification. The coal should be broken to the size of the fist, or smaller. The muffles are sufficiently hot for charging in from thirty to sixty minutes after the fire is lighted. The heat is easily regulated by means of the damper closing the ash-pit.

Many portable furnaces have been devised and are in use, such as the Hibbs, Brown, Battersea, etc. Oil and gas furnaces are used with varying results, but the limits of this work will not permit a description of them. See Mitchell's Manual of Practical Assaying, pages 71 to 107 incl.; also Brown's Manual of Assaying, pages 72 to 89, and the circular of the Buffalo Dental M'f'g Co.

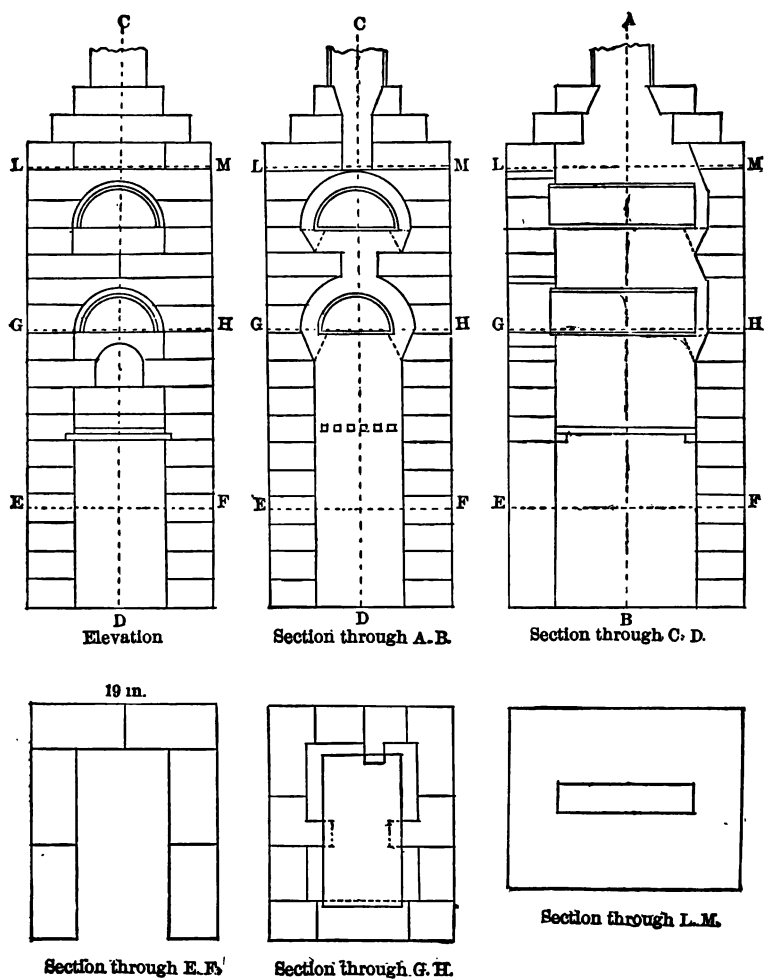


FIG. 8.

**ASSAY FURNACE FOR BURNING SOFT COAL.**

Muffles 8x15 inches.

Bind with  $1\frac{1}{4}$  inch iron.

Ash pit and coaling hole closed by sheet iron door, the latter resting on the binders.

THE FUELS employed are coke, anthracite, bituminous coal, and charcoal. Sometimes oil and gas are used for small laboratory furnaces.

The coke should be about egg size and free from sulphur. It is chiefly used in calcining or when a quick fusion is desired.

Charcoal, coke, anthracite, and bituminous coal may be employed for the muffle furnace. Bituminous coal should have, however, a special furnace. (See Fig. 8.)

Anthracite coal, stove size, is best adapted for the assayer's purpose, but charcoal may be used as a substitute for either coke or anthracite, when it can be had more cheaply; it gives a hot fire, and is easily regulated; but requires constant attention, and the pieces used should be of medium size.

In lighting a fire it will be found convenient to use pieces of cork or corncobs saturated with rosin, which burn well, are cheap, and save much trouble, as they take less room than paper and give no dirt.

To use, it is only necessary to light a small piece and place it upon a little kindling-wood in the bottom of the furnace, then put a few pieces of wood on top and add the coal after the wood has kindled.

## CRUCIBLES.

A good crucible should stand sudden changes of temperature, be infusible, impermeable, and not attacked by fused substances.

The crucibles in use may be arranged in the following order:

1. Black lead or graphite for fusing metals.

2. French clay. (Fig. 9.)

3. Hessian sand crucibles, round and triangular. (Fig. 10.)



FIG. 9.



FIG. 10.

#### 4. Charcoal-lined crucibles.

The most refractory crucibles are cut out of quick lime, or can be moulded from magnesia and chloride of magnesium; but the latter, however, are soft and not very strong.

The composition of the black lead crucibles is generally one to seven parts of refractory clay, and three to ten of graphite; but sand is sometimes used. If the crucibles contain too much silicious matter they are liable to be acted upon by the melted charge, or the bases contained in the coal around them, when in the fire.

These crucibles run in sizes from 1 to 400. The smallest holding from two to three ounces of metal. The next, four to six, and so on.

The demand is for two kinds, "steel" and "brass;" but they can be employed for melting all substances which are not oxidizing or strongly alkaline in their action.

French crucibles are made of Paris clay and fine sand, and rank among the best, but are more expensive than the Hessian. For melting charges which can be poured, they

are superior, as the crucible can be used again. The sizes run from 1 to 20, with covers to match.

The composition of the ordinary Hessian crucible is about three-quarters clay (German), and one-quarter sand. They are round and triangular, and run in regular sizes, viz: Small fives, large do., up to eights. "Halves," holding one-half gallon, and "ones," holding one gallon, with covers to match.

The charcoal crucible is made by lining an ordinary clay or Hessian crucible with a mixture of charcoal and molasses. The charcoal employed should be very fine, and only just enough molasses used to hold it together. The mixture is then packed into the crucible as tightly as possible, dried slowly, and bored out to any extent desirable.

Sometimes water and gum are substituted for molasses.

Fig. 11 represents three kinds of charcoal-lined crucibles.



FIG. 11.

Alumina crucibles for some operations are very satisfactory when intense heat is required, but lime will answer as well.

The choice of a crucible depends upon the nature of the substance to be treated in it, the temperature, and the time it is to remain exposed to the action of heat.

If a charge be basic the crucible should be basic also, and *vice versa*. The grain and appearance of the crucible should be taken into consideration. Much iron will render the crucible fusible.

To test a crucible for fusibility, heat a piece of the crucible and see if the corners are rounded, or if it is fused on the edges.

For corrosive action fuse litharge in the crucible. For permeability fill two crucibles with water and note the time required for it to run out, the one which holds the best being preferable. As a rule, crucibles resist permeation and corrosion in the proportion of the fineness and regularity of grain, but their tendency to crack is increased in the same ratio.

The action of sudden changes of temperature may be ascertained by heating suddenly, and cooling first in air and afterwards by plunging in cold water.

ROASTING DISHES (Fig. 12), and SCORIFIERS (Figs. 13

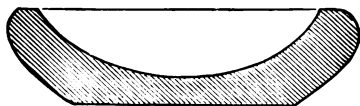


FIG. 12.



FIG. 13.

and 14). Both dishes and scorifiers are made of refractory clay the same as crucibles. They should resist the action of litharge and not be too deep. Painting with water and

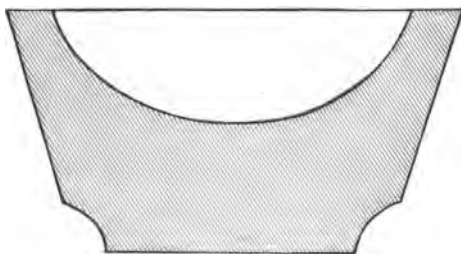


FIG. 14.

oxide of iron prevents, to some extent, the cutting by strong bases.

Scorifiers may be bought or made, but as a rule it is

better to buy them, as they will stand transportation and it requires some care to make good ones.

A section of a good scorifier should be uniform in character, close, and show no flaws or cracks. (Fig. 14.)

### CUPELS.

These vessels are generally made of the ashes of burnt bones, freed from organic matter, ground and washed. horse- or sheep-bones are said to be the best.

It is better to make cupels than to buy them, especially when they have to be carried some distance. The prepared bone-ash can be obtained in bulk, and is mixed with just sufficient warm water or stale beer, to cause it to hold together without being moist or wet when pressed into a ball in the hand.

The consistency of the mixture should be such that it will retain its shape when pressed, but crumble readily when disturbed.

Too much bone-ash should not be mixed at once, as it dries quickly.

If the bone-ash is too fine or too coarse it works badly; as in the first case the cupel will crack upon drying, and in the second, be too porous, absorb silver with the litharge and occasion loss.

The cupel is formed by filling and driving the prepared bone-ash into a mould made for the purpose.

The right degree of compression should be used, as otherwise the cupel will be either too hard or too porous.



FIG. 15.

A little experience will tell the operator when he has reached the proper point. When completed it presents the appearance of Fig. 15.

Care should be used in drying, plenty of time being allowed, and all moisture and organic matter expelled previous to using, by heating in a furnace. Sometimes a

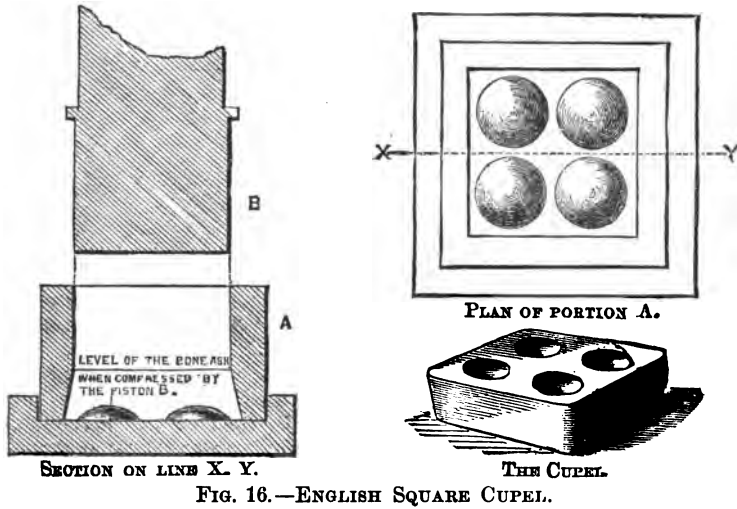


FIG. 16.—ENGLISH SQUARE CUPEL.

cupel is made of coarse bone-ash, and the surface finished off with fine washed material.

Cupels dried in the sun are better than those dried artificially. They are not so liable to crack.

In the Royal Mint, London, England, an improved form of cupel is in use. It is square, with four depressions for holding the same number of buttons, enabling the operator to run two assays in duplicate in the same cupel. Fig. 16 represents the method of making, and the cupel when finished. Iron-bound cupels are sometimes used when the amount to be cupelled is large, especially in treating sweeps. A good cupel can be made by pressing bone-ash into a muffin-ring.



## LUTES, CEMENTS, AND WASHES.

## GOOD FIRE LUTES.

1. Fire clay, two parts.

Sharp sand, eight parts.

Horse dung, one part.

Mix well and temper the same as mortar, until the desired consistency is reached.

2. Fire clay, one part.

Sand, three parts.

Mix with a little hair and weak borax water.

3. Zinc cement.

Dissolve borax in water to about 1.49 specific gravity and then add calcined oxide of zinc to suit.

## OTHER LUTES.

Plaster of paris mixed with water, milk, glue, or starch water makes a good lute, and will stand a red heat. Wax or paraffin is useful for bottles, stoppers, etc., also tallow or stearic acid. Faraday's cap cement is made by melting together rosin, five parts, yellow beeswax, one part, and stirring in one part of red ochre.

A fine lute for iron vessels is porcelain clay (kaolin) mixed with a solution of borax in water.

A good lute for glass vessels, is quicklime slaked in the air and then beaten into a liquid paste with white of egg.

Where corrosive vapors are liable to escape, a lute made of fire clay and boiled linseed oil should be applied, and covered with slips of linen spread with the lute of lime and egg.

### TO LINE CRUCIBLES.

Use fine sifted charcoal mixed with gum water, borax water, or molasses enough to hold together when pressed in the hand, without being wet or sticky. It should contain no lumps.

### WASH FOR CRUCIBLES AND SCORIFIERS.

1st. Finely pulverized chalk and water.

2d. Sesquioxide of iron (hematite) and water.

### TOOLS.

The tools required by the assayer are regulated more or less by the work to be done.

The following are the principal:

Crucible tongs (Fig. 17). They should be made with



FIG. 17.

long handles for taking crucibles out of the fire.

For heavy crucibles the shape shown in Fig. 18 is preferable.



FIG. 18.

Scorification tongs (Fig. 19). The spring should not



FIG. 19.

be too strong, and the horseshoe part should just fit the scorifier.

Cupel tongs (Fig. 20). These should be made of steel



FIG. 20.

and about two and one-half feet long with an easy spring.

Muffle tongs (Fig. 21), for handling small crucibles in the muffle

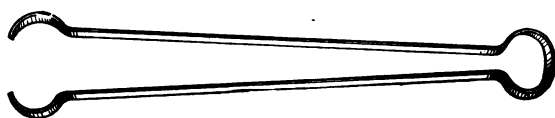


FIG. 21.

Three hammers will be useful. One large for hammering metal, one medium for breaking crucibles and scorifiers, and one small for marking lead buttons.

A set of small steel dies from 0 to 9 inclusive, and large and small alphabet for marking buttons and bullion will be found useful, but are not absolutely necessary.

Three pokers are desirable, small, large, and medium.

One or two small hoes or scrapers for cleaning out the bottom of the cupel muffle (Fig. 22).

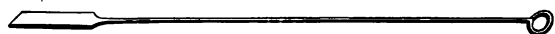


FIG. 22.

A pair of cutting shears and nippers for cutting wire for lead assay, etc.

A small vise and anvil, medium size, with a suitable bench for the same.

Wooden mallets, light and heavy, for packing crucibles, making cupels, etc.

Files and cold chisels for sampling and cutting metals. Charcoal saw for blow-pipe charcoal, and crucible tops.

Two iron mortars and pestles, and if much ore is to be pulverized, a grinding plate and rubber, as shown in Fig. 23, will be a great convenience and save labor. The plate

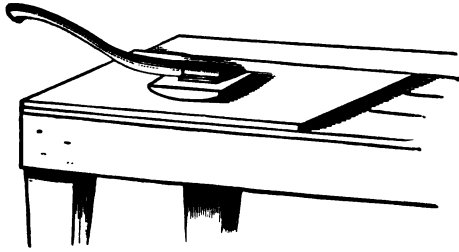


FIG. 23.

is a flat iron casting  $18 \times 24$  inches, and 1 inch thick, the surface used being planed smooth. The rubber or grinder is a piece of cast iron,  $4 \times 6$  inches square,  $1\frac{1}{4}$  inches in the middle, by  $\frac{7}{8}$  of an inch thick at the ends; thus giving a slightly convex surface, which should be true on the board at all points. To conduct the operation place the left hand upon the rubber, throwing the weight of the body upon it, and then grasping the handle with the right hand, move the iron rubber back and forth, depressing the handle when pushing forward and raising it in drawing back. For laboratories where large quantities of ore are to be pulverized, the size of the plate, and the weight of the rubber should be increased; but for ordinary use the dimensions given will be found sufficient. Too much ore should not be placed upon the plate at a time, as it makes the operation of pulverization more difficult.

The operation is much more rapid than with the ordinary mortar and pestle, and the manipulator after a little practice has complete control over the ore treated.

Should it not be convenient to use the plate and rubber, a long handled pestle coming up to the chest will be found an improvement, as the mortar can be placed on the

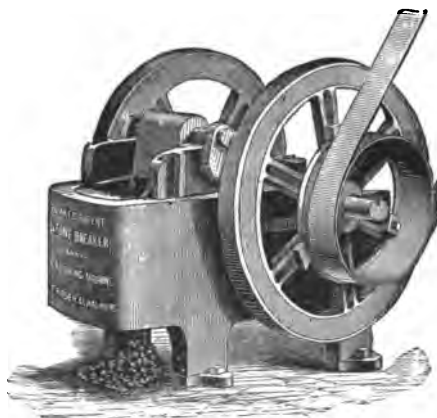


FIG. 24.—BLAKE CRUSHER.

floor and the pestle worked while the operator is in a standing position.

Where large quantities of ore are to be ground a



FIG. 25.—PULVERIZER.

crusher and a pulverizer run by power will be found necessary (See Figs. 24 and 25).

A series of sieves, from twenty to one hundred mesh, will be useful for sifting ores and fluxes. The box sieve (Fig. 26), is a simple arrangement, and consists of a round tin box with a sieve fitting into it as represented in the illustration. The sieve is a tin frame with any desired mesh gauze soldered to it, and fits tightly in the box. The advantage gained by its use is that in sifting the pulverized ore there is no dust, the fine material after passing through the sieve is kept from flying around. The size most convenient is 8 inches in diameter, the box 2 inches deep, and the rim of the sieve 2 inches, fitting about  $\frac{1}{4}$  inch into the box. A tin cover can be placed over the whole.



FIG. 26.

Open and closed ingot moulds for casting lead and silver bars, ingots, etc.



FIG. 27.

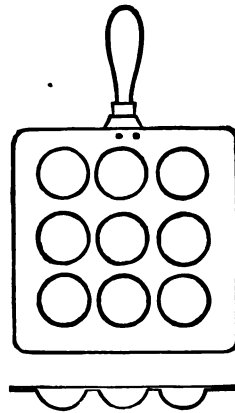


FIG. 28.

Hand button-rolls for gold and silver only. They should be kept covered and free from dust.

Cupel mould (Fig. 27). This consists of two parts, an

iron ring and a steel pestle or driver, just fitting into the ring.

A mould for pouring the assay charge in scorification (Fig. 28). This should be of heavy cast iron or copper. It saves much time, and by employing it, the scorifiers can be used again. Larger moulds of the same style will be found convenient for pouring crucible charges, but are not necessary, unless crucibles are scarce.

Shovels for coke and coal, and a small hatchet for splitting kindling wood. The coke shovel should be ribbed or perforated so that the fine coke or dust may fall through.

Mixing scoops of Russia sheet iron  $3\frac{1}{2}$  by 5 inches, with straight sides and back about  $\frac{3}{4}$  inch high. They are convenient for mixing lead or silver crucible charges in, and owing to the high finish of the iron, the assay on being poured out does not cling to the scoop, a few sharp taps detaching everything.

A tin sampler, shown in Fig. 29, will be found very useful. It consists of a series of troughs arranged in a row and fastened together at equal distances by a tin strip soldered on their ends. A shovel full of ore emptied by a series of shakes across them, is just half caught by the troughs; one-half going through the openings between. By repeating this operation, the size of the sample can be reduced to any extent desired.



FIG. 29.

Mechanical samplers are much used in the large establishments in the West, and are recommended when large lots of ore are to be treated. There are many kinds in use, but all depend on separating mechanically a fractional part of a falling quantity of ground ore. Their

chief objection aside from expense is the difficulty in thoroughly cleaning them.

Fig. 30. shows the Bridgman ore-sampling machine.

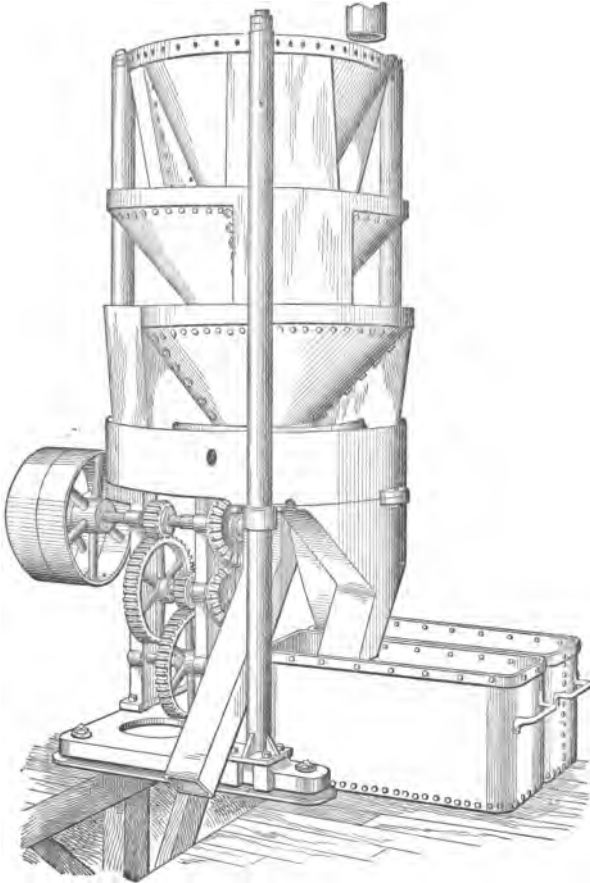


FIG. 30.—BRIDGMAN ORE-SAMPLING MACHINE.

In passing through this machine the fall of the ore is partially intercepted by three revolving apportioners. The uppermost of them separates two parts, each from one-half to one-eighth of the whole lot, according to adjustment.



These parts are successively quartered by each of the lower apportioners. This division is accomplished at a low rate of speed by giving each of the lower apportioners three times the revolutions of the one above it, and causing the middle apportioner to revolve in the reverse direction. The maximum capacity of the machine is 20 tons per hour. Even if the feeding is irregular the samples give satisfactory results. For a detailed description of this machine see "A New System of Ore Sampling,"

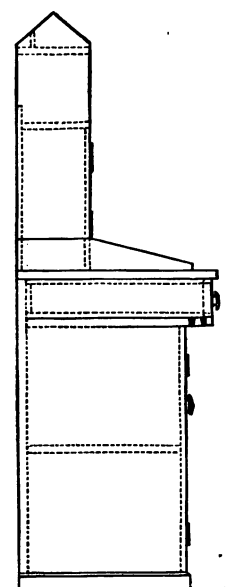


FIG. 31.—SECTION.  
Scale  $\frac{1}{4}$  inch to the foot.

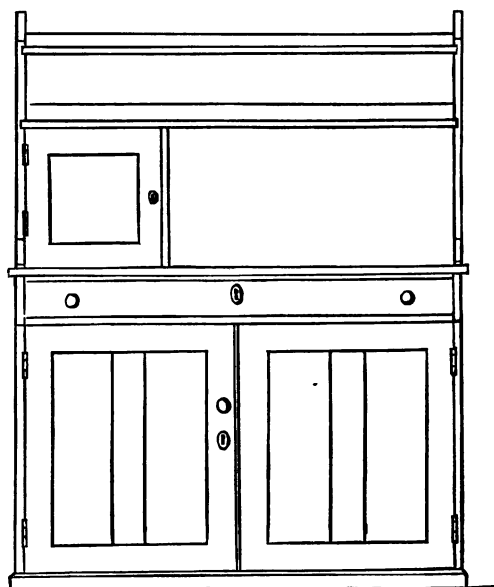


FIG. 32.—ELEVATION.  
Scale  $\frac{1}{4}$  inch to the foot.

by H. L. Bridgman: Trans. Am. I. of M. E. Oct. 1891. Also Hofman's "Metallurgy of Lead," and Peters' "Modern Methods of Copper Smelting," 7th Edition.

A laboratory desk, as shown in Figs. 31 and 32, will be found a very suitable and compact arrangement. It con-

sists of four parts, shelves for bottles, closet for ore-scales, drawer for cupels and apparatus, and double closet for crucibles, scorifiers, etc. The illustrations are made to a scale so the desk can be constructed from them without trouble.

This style of desk has been in use in the School of Mines, New York, for many years, and has been found most convenient. The lower closet should be provided with a shelf and the drawer with partitions. If gas can be had, each desk in a laboratory should have a burner above for lighting purposes, and two or three large jets to which rubber tubes can be fastened so that Bunsen burners can be employed on the desks. These jets are best placed next the scale closet.

## APPARATUS.

The amount and kind of apparatus required by the assayer varies, but in the following list will be found all that will be needed for ordinary work :

About three dozen quart bottles for reagents, glass stoppered. One dozen glass-stoppered parting bottles, for bullion assay. Eight oz. is a good size. The stoppers should be square-topped and fit exactly, so that the bottles will not leak when shaken.

An assortment of corked bottles of different sizes for samples.

Two or three ring stands and the same number of Bunsen burners or alcohol lamps. The former are preferable, if gas can be had, and should be provided with two or three feet of rubber tubing for each burner.

Two wash bottles, one small and one large, say one-half pint and quart.

One half-dozen horn spatulas or spoons for mixing and some iron pans for roasting ore. The ordinary long-handled frying pan is suitable, and should be about the size of the furnace top. Before roasting it should be lined with chalk or oxide of iron. One dozen parting flasks (Fig. 33), for gold bullion assay; also annealing cups for the same purpose. These are of clay and made thin (Fig. 34).



FIG. 33.



FIG. 34.

Brushes for ores and fluxes made of camels-hair; a large feather trimmed, makes an excellent substitute.

A few dozen sheets of glazed paper, or stout manilla paper when glazed paper cannot be had, for mixing ore upon. Black is preferable, and when held up to the light, there should be no holes.

Hessian and French crucibles and covers of various sizes and shapes.

Scorifiers, large and small.

Scorification and cupel muffles to suit furnaces.

Cupels from  $\frac{3}{4}$  to 2 inches in diameter.

These should always weigh more than the button to be cupelled.

For volumetric work, silver bullion, etc., graduated flasks, pipettes and burettes, will also be necessary. See bullion assay, page 146.

For the volumetric and gravimetric methods of analysis (see Part III) the following apparatus will be necessary:

Reagent bottles (glass stoppers).

Funnels of various sizes.

Glass tubing.

Glass rods of different sizes.

Test tubes and rack.

Set of graduated flasks 100 c.c., 200 c.c., 250 c.c., 500 c.c., and 1 litre. These flasks should have narrow necks, to give greater accuracy in filling, and are best when provided with ground glass stoppers.

Erlenmeyer flasks, 100 and 250 c.c.

Pipettes 10, 50, and 100 c.c.

Convex covers—12 of various sizes.

Measuring cylinders 10 c.c. and 100 c.c.

Beakers, 3 nests lipped.

Dessicator, 1 medium size.

Burettes, Mohr stop cock, or pinch, with float or blue back and stand.

It is advisable to have one burette for each solution in constant use.

Casseroles (Royal Berlin), 6.

Cut filters (ashless), 3 sizes.

Gummed labels.

Ring stands, 2 (3 rings each).

Files, round and triangular.

Forceps for small crucibles.

Bunsen burners, 3, and clay chimney; or if gas is not to be obtained, small kerosene stoves, and 1 small alcohol lamp.

Tin or Russia-iron sand baths.

Asbestos discs.

Filter stands, Fresenius, 4.

Rubber tubing (assorted).

Sponge swab, tube cleaner, towels, etc.

Agate and porcelain mortars with pestles.

Crucibles, porcelain of 3 sizes ; platinum, 1 of 20 gms. and 1 of 40 gms.

Triangles, 1 platinum, 2 pipe-stem.

Thermometer, centigrade.

Battery cells, 3 or 4, Edison-Laland, or gravity, etc.

Platinum electrodes and stand.

## REAGENTS AND CHEMICALS.

Reagents for fire assays :

These may be divided into seven classes.

*a.* Reducing. To this class belong those bodies which have the power of removing oxygen from its combinations.

*b.* Oxidizing. All bodies which give up oxygen with facility. Used in roasting and crucible work.

*c.* Desulphurizing. This class includes all substances which possess a strong affinity for sulphur, and will decompose its compounds under the action of heat or in solution.

*d.* Sulphurizing. Sulphur and such of its compounds as give up their sulphur easily upon elevation of temperature or in solution.

*e.* Fluxes. Under this head, we include a large class of bodies, either acid, basic, or neutral in their action.

They are used—

1st. To cause the fusion of substances otherwise infusible, by forming new combinations.

2d. To fuse or hold in suspension foreign substances mixed or combined with a metal, so as to allow the metal to separate and settle by its greater weight.

3d. To obtain a metal in one button and not in globules or prills.

4th. To prevent the formation of alloys.

*f.* Solvents generally include solutions which are used in the wet way only, such as distilled water, nitric, sulphuric, and hydrochloric acids, etc.; but some substances, such as metallic lead, etc., used in crucible work are sometimes classed as solvents.

*g.* Precipitants in the wet way. As the salt solution used in the bullion assay.

The following are the principal reagents and chemicals employed by the assayer in his work. There are, however, many others which might be used, but they can all be classed under the heads just given.

Dry BICARBONATE OF SODA (sodium bicarbonate,  $\text{NaHCO}_3$ ) or potassium carbonate ( $\text{K}_2\text{CO}_3$ ). These act as desulphurizing agents, and in some cases as oxidizing agents. The latter action is due to the carbonic acid contained. They also act as basic fluxes.

They should be free from moisture and lumps. On account of their easy fusibility they can retain in suspension, without losing their fluidity, a large proportion of finely powdered infusible substances.

LITHARGE ( $\text{PbO}$ ), is a basic flux, oxidizing and desulphurizing agent, and supplies the lead in the gold and silver crucible assay. It should be dry, and free from red oxide of lead, as the latter has the power of oxidizing silver, and thus causing loss of that metal during the assay. To free litharge from the red oxide, fuse the same in a crucible, and pour the mass into a cold ingot-mould, keeping it from the air while cooling. All litharge before

using should be well sampled, and assayed for silver. To do this, charge in a crucible—

|                                |          |
|--------------------------------|----------|
| Litharge.....                  | 4 A.T.   |
| Soda ( $\text{NaHCO}_3$ )..... | 2 “      |
| Argol .....                    | 2 grams, |

and cover with a layer of dried salt, one-quarter of an inch thick. Fuse in a hot fire until completely liquid, then withdraw, and proceed as in the assay of a silver ore (p. 99). White lead (basic carbonate of lead) or plumbic carbonate ( $\text{PbCO}_3$ ), is sometimes employed instead of litharge; also, acetate of lead for delicate experiments.

**BORAX, CRYSTALLIZED** ( $\text{Na}_2\text{B}_4\text{O}_7, 10\text{H}_2\text{O}$ ). This acts as an acid flux; but, on account of the water contained, it is generally employed in a vitrified condition, or borax glass ( $\text{Na}_2\text{B}_4\text{O}_7$ ), which has a more intensified effect. It has neither an oxidizing nor desulphurizing action. It is sometimes used as a cover in place of salt.

To prepare—Fuse the commercial borax in a large sand crucible and pour the fused mass out on a clean surface to cool. Pulverize, and keep in a glass-stoppered bottle. As borax, when heated, loses its water of crystallization, and undergoes an immense increase in volume, only a little should be added at a time in fusing. Boracic acid ( $\text{H}_3\text{BO}_3$ ) is also sometimes employed.

**SILICA** ( $\text{SiO}_2$ ), acts as a good acid flux, and can often be used with advantage. A good substitute is glass ( $\text{Na}_2\text{Si}_3\text{O}_7 + \text{CaSi}_3\text{O}_7 + \text{SiO}_2$ ), as it is easily fusible, and forms a good slag. It should be powdered and free from moisture. Lime glass is the best.

**BLACK FLUX, SUBSTITUTE.**—A mixture of three parts flour and ten parts of sodium bicarbonate. It acts as a

flux and reducing agent, and is especially useful in the lead assay. Black Flux, proper, is 1 part of nitre and 3 parts of argol deflagrated.

LEAD FLUX.—A mixture of 4 parts sodium bicarbonate ( $\text{NaHCO}_3$ ), 4 parts potassium carbonate ( $\text{K}_2\text{CO}_3$ ), 1 part flour, and 2 parts borax glass. Its action is reducing, desulphurizing, and fluxing.

MODIFIED LEAD FLUX.—Sodium bicarbonate 2 parts, potassium carbonate 2 parts, borax glass 1 part, argol 1 part.

CYANIDE OF POTASSIUM (potassium cyanide,  $\text{KCy}$  or  $\text{KCN}$ ), as a flux for reducing and desulphurizing is invaluable. It is also employed in solution as a solvent for gold, and in the amalgamation assay. It should be prepared with care and kept in a tight bottle, as it absorbs moisture. Take the ordinary commercial article and pulverize in an iron mortar as fine as possible. Never sift, as the dust is *poisonous*. To protect yourself, cover the mortar with a towel, or a board having a hole in the centre for the pestle.

ARGOL ( $\text{KHC}_4\text{H}_4\text{O}_6$ ), crude bitartrate of potassium, acts as a basic flux and reducing agent. It should be pulverized and dry, and its reducing power determined. For this purpose we charge

|               |                 |
|---------------|-----------------|
| Argol .....   | 2 gms.          |
| Litharge..... | 2 A.T.          |
| Soda .....    | $\frac{1}{2}$ “ |

in a crucible, fuse in a hot fire, cool, extract the button and weigh in grams. Dividing by two gives the amount of lead one gram of argol will reduce from litharge.

CHARCOAL, acts as a reducing or deoxidizing agent.



It should be finely powdered and its reducing power determined, as in the case of argol. Use charcoal one gram, litharge 2 A.T., and soda  $\frac{1}{2}$  A.T. Ordinary wood charcoal will reduce twenty-eight grams of lead from litharge.

STARCH, flour, sugar, and gum, may also be used as reducing agents, but are not so convenient.

Dried starch reduces thirteen parts of lead. Common starch about eleven and one-half parts. Sugar fourteen and one-half, and gum arabic eleven parts. For some purposes pure hydrogen gas will be found useful. One part of hydrogen will reduce about one hundred and four parts of lead from litharge.

METALLIC IRON (Fe), is a desulphurizing agent, and is indispensable, especially in the assay of lead ores. The best form is iron wire about  $\frac{1}{8}$  inch in diameter. Nails and filings may also be used.

PURE LEAD (Pb), in sheet or granulated form, is used principally in the assay of silver ores. It acts as a basic flux, and a solvent or wash for the precious metals. The sheet form is useful in cupelling gold and silver beads, and in the bullion assay. The granulated is essential in the scorification assay.

It can be obtained pure by decomposing the best white lead by charcoal, and granulating or fusing in bars, as the case may require.

The desilverization works now produce pig lead containing only about 10 cents in silver per ton, which is quite pure enough for all ordinary assay work.

In sections where granulated lead free from silver, or white lead, cannot be obtained, the assayer can make his own granulated lead from such pig lead, poor in silver, by

the following method :—Melt about fifty pounds of lead in an iron pot or crucible, and keep it just at the melting point. Then pour a ladleful of the melted lead into a wooden bread-tray which has been dry chalked on the inside. Keep this agitated by gently rocking the tray to prevent solidification, and when the mass begins to get pasty, throw it into the air and catch it again repeatedly until cold, when the metal will be found to be nearly all granulated. Sift through a twenty-mesh sieve, and what does not go through re-melt. The whole fifty pounds can be granulated in this way in two hours. After granulation sample well and test eighty or one hundred grams for silver, by the scorification assay. In using the lead, the silver contained in it must be deducted from the results obtained in assaying an ore.

PURE SILVER (Ag), in sheets for inquarting buttons and gold bullion. Where silver foil cannot be obtained pure, it can be made from silver coins or bar silver by dissolving in nitric acid, filtering off any trace of gold which may be present, precipitating the silver as chloride, and then reducing the chloride to metallic silver with zinc and sulphuric acid and after washing, melting the silver with a little nitre and borax in a graphite crucible.

NITRE (potassium nitrate,  $\text{KNO}_3$ ), acts as an oxidizing agent and also as a basic flux. It should be finely powdered, dry, and assayed for its oxidizing power. Charge:

|                |                 |
|----------------|-----------------|
| Nitre.....     | 3 gms.          |
| Charcoal.....  | 1 “             |
| Litharge. .... | 2 A.T.          |
| Soda .....     | $\frac{1}{2}$ “ |

Place in a Hessian crucible and cover with salt. Fuse in a hot fire, remove, cool, and weigh. The difference between the weight of the button obtained and that given in the assay of charcoal, divided by three, gives the oxidizing power of nitre per gram.

POWDERED LIME ( $\text{CaO}$ ) (dry) and fluor spar ( $\text{CaF}_2$ ), will often be found useful as basic fluxes, especially in the assay of iron ores. Magnesia ( $\text{MgO}$ ), and alumina ( $\text{Al}_2\text{O}_3$ ) or kaolin ( $\text{Al}_2\text{O}_3, 2\text{SiO}_2$ )—are also used, and cryolite ( $3\text{NaF}, \text{AlF}_3$ ) for special cases.

AS SULPHURIZING AGENTS, powdered sulphur ( $\text{S}$ ), pure galena ( $\text{PbS}$ ), or sulphide of antimony ( $\text{Sb}_2\text{S}_3$ ), are employed.

. CARBONATE OF AMMONIA (ammonium carbonate,  $(\text{NH}_4)_2\text{CO}_3$ ), as a desulphurizing agent, is used in the decomposition of some sulphates, as sulphate of copper, in roasting. It should be powdered and kept in a closed vessel.

COMMON SALT (sodium chloride,  $\text{NaCl}$ ), as a cover and wash, and as a reagent in the bullion assay, should be always kept on hand. The purer it is the better, and it must also be fine and dry.

METALLIC ARSENIC ( $\text{As}$ ), as a collecting and fluxing agent in special assays, such as nickel, copper, etc.

As solvents, distilled water ( $\text{H}_2\text{O}$ ), nitric acid ( $\text{HNO}_3$ ) sp. gr. 1.16 and 1.26, free from chlorine, hydrochloric acid ( $\text{HCl}$ ), sulphuric acid ( $\text{H}_2\text{SO}_4$ ), sodium thiosulphate ("Hypo,"  $\text{Na}_2\text{S}_2\text{O}_3, 5\text{H}_2\text{O}$ ) are used.

CHLORINE ( $\text{Cl}$ ), generated from bleaching powder and sulphuric acid, or from salt, manganese dioxide, and sulphuric acid, is employed as a solvent for gold in special tests.

## REAGENTS FOR WET ASSAYS.

*a.* Acids:

Acetic ( $\text{HC}_2\text{H}_3\text{O}_2$ ), chem. pure 50%.

Hydrochloric ( $\text{HCl}$ ), chem. pure concentrated 1.2 sp. gr ; dilute 1 part concentrated to 2 parts water.

Nitric ( $\text{HNO}_3$ ), chem. pure concentrated 1.42 sp. gr.; dilute 1 part concentrated to 2 parts water.

Oxalic ( $\text{H}_2\text{C}_2\text{O}_4, 2\text{H}_2\text{O}$ ), chem. pure crystals.

Sulphuric ( $\text{H}_2\text{SO}_4$ ), chem. pure concentrated 1.82 sp. gr.; dilute 1 part concentrated to 4 parts water, also common dilute for making sulphuretted hydrogen.

Sulphurous ( $\text{H}_2\text{SO}_3$ ), chem. pure solution.

Tartaric ( $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$ ), chem. pure powder.

Hydrosulphuric acid or sulphuretted hydrogen, ( $\text{H}_2\text{S}$ ) is best prepared from pieces of sulphide of iron (ferrous sulphide,  $\text{FeS}$ ) and dilute sulphuric acid, the gas being passed through a second bottle filled with water to wash it. Fig. 31 shows the apparatus in position for use. The glass tubes are connected with small pieces of rubber tubing. The gas may be passed into the solution to be precipitated, or a water solution may be saturated, and used at pleasure. The sulphide of iron can be made by heating scrap iron or borings to a red heat in a crucible and throwing in sulphur. The sulphide produced may then be fused or broken up ; complete fusion is unnecessary.



FIG. 31.

Hydrofluoric acid (HF), chem. pure in Ceresine bottles (Baker & Adamson).

The following reagents should be chemically pure, unless specially noted.

*b.* Alkalies and alkaline salts.

Ammonia ( $\text{NH}_4\text{OH}$ ), aqueous solution, sp. gr. 0.9.

Sodium hydroxide ( $\text{NaOH}$ ), 10% solution.

Sodium carbonate ( $\text{Na}_2\text{CO}_3$ ), pulverized and dry for fusions.

Sodium carbonate, cryst. ( $\text{Na}_2\text{CO}_3, 10\text{H}_2\text{O}$ ), saturated solution.

Potassium hydroxide ( $\text{KOH}$ ). 10% solution.

Potassium carbonate ( $\text{K}_2\text{CO}_3$ ), anhydrous and pulverized.

Fusion mixture, 14 parts  $\text{K}_2\text{CO}_3$  and 11 parts  $\text{Na}_2\text{CO}_3$  pulverized and thoroughly mixed.

*c.* Salts.

Ammonium chloride ( $\text{NH}_4\text{Cl}$ ).

Ammonium molybdate ( $(\text{NH}_4)_2\text{MoO}_4$ ), see page 182.

Ammonium nitrate ( $\text{NH}_4\text{NO}_3$ ).

Ammonium hydrogen sulphide ( $\text{NH}_4\text{HS}$ ), made by passing  $\text{H}_2\text{S}$  into dilute ammonia.

Ammonium sulphide yellow, made by dissolving an excess of sulphur in the colorless (acid) sulphide.

Ammonium sulphocyanide ( $\text{NH}_4\text{CNS}$ ), dilute solution.

Ammonium acetate ( $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$ ).

Sodium bromide ( $\text{NaBr}$ ). Sodium chloride ( $\text{NaCl}$ ).

Sodium acetate ( $\text{NaC}_2\text{H}_3\text{O}_2$ ) 10% solution.

Sodium nitrate ( $\text{NaNO}_3$ ).

Sodium ammonium hydrogen phosphate ( $\text{NaNH}_4\text{HPO}_4, 4\text{H}_2\text{O}$ ). (Salt of phosphorus or microcosmic salt.)

Sodium thiosulphate ("Hypo,"  $\text{Na}_2\text{S}_2\text{O}_3, 5\text{H}_2\text{O}$ ).

Potassium bichromate ( $K_2C_{22}O_7$ ).

“ bisulphate ( $KHSO_4$ ) (fused).

“ chlorate ( $KClO_3$ ).

“ ferrocyanide ( $K_4FeCy_6, 3H_2O$ ) (yellow prussiate).

“ ferricyanide ( $K_3FeCy_6$ ) (red prussiate).

“ permanganate ( $K_2Mn_2O_8$ ).

“ iodide (KI).

Barium chloride ( $BaCl_2, 2H_2O$ ) 10% solution.

Barium carbonate ( $BaCO_3$ ), free from ammonia salts.

Magnesia mixture, made by dissolving together 1 part magnesium chloride, 2 parts ammonium chloride in 8 parts water, then adding 4 parts of dilute ammonia, and filtering after several days.

Silver nitrate, ( $AgNO_3$ ). This can be prepared by dissolving pure silver in nitric acid.

Platinic chloride or hydrochloroplatinic acid ( $H_2PtCl_6$ ), strong solution.

Cobalt nitrate ( $Co(NO_3)_2, 5H_2O$ ).

Uranium acetate ( $(UO)_2(C_2H_3O_2)_2$ ).

Stannous chloride ( $SnCl_2, 2H_2O$ ).

Mercuric chloride ( $HgCl_2$ ).

Ferrous sulphate ( $FeSO_4, 7H_2O$ ) free from phosphorus.

*d. Metals.*

Zinc, sheet and granulated, free from arsenic and containing but a trace of iron.

Aluminium sheet. Copper, electrolytic.

Lead, test lead. Iron, wire, 99.7% Fe.

Mercury, redistilled.

*e. Miscellaneous.*

Magnesium oxide ( $MgO$ ), free from sulphur if possible, or with the percentage of sulphur accurately determined.

Zinc oxide (ZnO). Bromine (Br). Alcohol (ethyl) ( $C_2H_5OH$ ), 95%. Starch ( $C_6H_{10}O_5$ ).

Methyl orange, and litmus as indicators.

### PRELIMINARY TESTING OF ORES.

Before breaking up a sample it should be thoroughly examined to determine, if possible, its mineralogical character. When the sample submitted is already ground, a small portion should be panned down on a large watch glass and the concentrates, consisting of the heavy minerals, sulphurets, native metals, etc., carefully examined with a magnifying glass.

If this operation is done carefully it often determines the character of the ore with sufficient accuracy to enable an experienced assayer to select a method of assay and make up the correct charge.

A fusion of ten grams of ore with forty of potassium cyanide will show the presence of lead, antimony, tin, copper, bismuth, large quantities of silver, and iron if the heat is high.

If the mineralogical character of the gangue cannot be determined by inspection, test for carbonates by nitric acid. Should the preceding tests be insufficient, try the blowpipe tests described in Part IV.

### SAMPLING AND PULVERIZING.

The selection and preparation of the sample for assay may be called the "secret of success." It is the most im-

portant operation which the assayer has to conduct ; and unless the sample be well taken his work will be useless.

No matter how large or how small the amount of ore he may be called upon to treat, the same care is necessary in the sampling ; for one portion may be very rich and another portion valueless, so far as the metal sought for is concerned. The sample, therefore, taken for an assay must *always* be an *average* of all the ore.

The method of sampling an ore depends upon its constitution :

- a. The ore contains no metallic particles.
- b. The ore contains metallic particles.

In the first case the operation is comparatively easy. If there is a large quantity of ore to be sampled, it is broken up more or less finely, the degree of fineness depending upon the amount of ore from which the lot for assay is to be taken, and is then either thrown upon a sampler (page 32) or divided by piling it in a heap and cutting it into quarters, two of which may be selected to be again broken up, mixed and quartered, and so on until a sample sufficiently small for assay is obtained ; or an equal portion of each quarter may be taken, and the four portions well mixed, broken up, thrown in a heap, and the operation repeated until the required sample is reached. A single specimen or the lot obtained by sampling as above, should be crushed and all passed through an eighty or one hundred mesh sieve—the finer the better. The pulverized ore is then well mixed with a spoon or spatula on glazed paper, and the amount for assay weighed out by taking a little here and there, or dividing into quarters and taking some from each quarter. As already stated, various mechanical devices are em-



ployed for sampling down large quantities of ore, but for most purposes the tin sampler described will be found sufficient. The size of this may be increased if desirable.

The fine ore should never be shaken to mix it, or poured upon the scale-pan directly from the vessel in which it is contained.

The entire contents of the bottle containing sample to be assayed should be emptied on glazed paper, mixed thoroughly, spread out thin, and portions for assay taken at intervals with a spatula.

*b.* The ore contains metallic particles.

The sample may be selected from the heap of ore in the same manner as described under *a*, but a larger lot must be taken for assay and the whole pulverized, by pounding in an iron mortar and passed through an eighty-mesh sieve, which will divide the ore into two portions :

1st. Siftings.

2d. Metallic residue.

The siftings must be well mixed and sampled upon glazed paper, as just described.

The metallic residue must be tested as a whole and *not* sampled, or if the amount is large, can be fused with pure lead, and a weighed portion of the resulting alloy assayed.

The method of making the assay and calculation of results will be given hereafter.

Care must be taken in preparing a sample that all apparatus employed is clean, especially the mortars and sieves.

The first can be cleaned by pulverizing a little sand in them, or using a pumice-stone pestle, and the latter by rubbing with a clean towel or rapping upon a bench.

The box sieve (page 31) will be found very convenient, and better than the ordinary kind, as it prevents the loss

of dust which would alter, more or less, the value of the sample. The sieve should be used for nothing but ores, and carefully cleaned after each operation.

In general, the finer the ore is ground the better the sample; the richer the material the more difficult to sample.

Base Bullion should be sampled by drilling half through the bar at intervals from both sides, as the contents of precious metals may be very irregular, especially if the bullion contains zinc. The drillings, if brittle, may be ground fine in a coffee-mill and the portion for assay taken out with a spatula after thoroughly mixing the fine particles; or grinding in the coffee-mill may be omitted, and the coarse drillings may be weighed and remelted carefully to prevent either volatilization or oxidation, cast, and the bar sampled as before for assay. A steel punch cutting out a  $\frac{1}{4}$ -inch core may be used instead of a drill.

Pig copper is sampled in the same way, but not always remelted. The slag from remelting should be tested and the contents allowed for.

Gold or Silver Bullion, if very pure (nearly 1000 fine), is sampled by chipping from alternate corners above and below. It is usually necessary to remelt: take samples from first and last pourings, and granulate by pouring into water. To sample coins: for silver, stamp out small pieces from the centre and edge; for gold coins, cut slips running from the centre to the circumference. The coins can also be rolled out thin, and the slips cut up fine or portions stamped out for assay.

• Sometimes lead chips or granulated alloys are submitted to the assayer, which call for great care in sampling. Lots of this kind should be weighed and melted in a clean

crucible, carefully poured so as to save all the scum, the bar weighed and sampled by chipping or boring, and the scum weighed and scorified. The value of the original alloy per ton can then be calculated in the same way as an ore which contains metallic particles.

When the assayer has access to the melted bullion, dip samples can be taken from the centre of the pot after stirring.

### WEIGHING ORE AND REAGENTS.

The ore, litharge, test lead, oxidizing and reducing agents should be weighed accurately.

The ordinary fluxes may be weighed approximately; still it is better to weigh closely, as more uniform results are obtained.

The same pan of either the flux or ore balance should always be used for the weights, and the latter must be handled with the pincers provided for that purpose. The ore scales should be kept free from dust, and be adjusted before each weighing; for, next to the sampling, the weighing of the ore is most important.

When a number of assays of the same ore are to be made, weigh out the fluxes first, and then add the ore to each charge. In this way the work will be greatly facilitated.

Instead of weighing the pure granulated test lead, it can be measured. A very simple and good test lead measure is a graduated glass tube about  $\frac{1}{2}$  inch in diameter with a cork plunger fitted to slide up and down, or a test tube graduated for known weights. As far as possible glazed paper or watch glasses should be used in weighing, to pre-

vent substances from touching the scale pan—especially when using the quantitative analytical balance.

If the substance is one which is liable to absorb moisture from the air it should be weighed between watch glasses, fastened with a clip. Cyanides must never be weighed upon the pan direct.

The balance pans of the bullion or quantitative balance should never be handled with the fingers or set upon a rough surface.

When one kind of ore is to be frequently assayed, make up a large quantity of the correct flux, litharge, reducing agent, etc. This mixture can be measured out by a scoop holding the right quantity, and in this way much labor and time saved.

## DRYING, CALCINATION, AND ROASTING.

In drying the object is to drive off moisture; in calcination, carbonic acid and other volatile constituents; while in roasting the operation is conducted in such a manner as to ensure oxidation, and the elimination of sulphur, arsenic, antimony, etc. To dry or calcine a substance it is not necessary that the air should have free access, or that the material treated be constantly stirred. A high temperature is seldom necessary for drying, 100° to 120° C. being generally sufficient, but a white heat is sometimes required for calcination. To conduct the operation, crucibles and dishes will be found the most convenient vessels.

For roasting, combustion must take place, and consequently the vessels employed must be open and flat to allow the oxygen of the air to act freely. The ore must

be stirred continually, and when easily fusible, be mixed with some substance to prevent agglutination. Charcoal, graphite, or sand may be used for this purpose. The heat should be low at first, and raised toward the end of the operation; and in some cases chemicals mixed with the mass hasten the process, and render it more complete, as in the addition of carbonate of ammonia in roasting copper ores, which decomposes any sulphates which may have been formed. The operation may be performed on a crucible furnace in an iron pan lined with chalk or oxide of iron; or in an open vessel like a scorifier (Fig. 12), in a muffle furnace. In any case the draft of air should be strong, as the fumes are injurious; the ore, however, must not be blown out.

A very nice stirrer for this operation can be made from a piece of ordinary iron wire, by doubling it and bending down the loop like a small hoe, the ends of the wire being twisted together to form a handle.

## REDUCTION AND FUSION.

Reduction is the changing of an atom to a lower valence, as  $\text{CrO}_3$  to  $\text{Cr}_2\text{O}_3$ ,  $\text{Fe}_2\text{Cl}_6$  (Ferric Chloride) to  $\text{FeCl}_2$  (Ferrous Chloride), etc.; or to the element itself, as  $\text{PbO}$  (Litharge) to  $\text{Pb}$  (Lead). Usually it is understood to mean the removal of oxygen from the body acted upon. In assaying, reduction generally means the separation of a metal.

The operation of reduction is usually accompanied by fusion, which is simply melting, although they may act independently of each other. We have reduction without fusion, when oxide of copper ( $\text{CuO}$ ) is reduced to metallic

copper by hydrogen or alcohol ; while fusion without reduction often takes place, as in a muffle under ordinary conditions. Reduction and fusion are carried on in crucibles, scorifiers, etc.

The heat required is higher than that necessary for the foregoing operations ; consequently the draft should be stronger. Fusion is sometimes a preliminary step to oxidation and sublimation.

To perform the operation of reduction in a muffle furnace, the muffle must be partially filled with charcoal, and the mouth closed.

## DISTILLATION AND SUBLIMATION.

Distillation usually means the conversion of a liquid into vapor by means of heat, and subsequently reconvertng to the liquid condition either with or without a condenser; it should, however, also apply to solids which pass through the liquid state before or after vaporization, as here it is the liquid which is really distilled, though the final product is solid.

Distillation is used as a process of purification or of separation ; for example, mercury after cleaning is redistilled to leave behind any non-volatile impurities. Arsenious chloride may be separated from antimonious chloride by distilling. Sublimation is similar to distillation; but the product, at the ordinary pressure, passes directly from the gaseous to the solid state; for example, white arsenic ( $\text{As}_2\text{O}_3$ ) is sublimed, not distilled.

Both operations may be conducted in flasks, retorts, or

crucibles ; but usually in the operation of distillation a cooled condenser is necessary, as in the process of making distilled water. The term "destructive distillation" is used where the body acted upon undergoes decomposition.

### SCORIFICATION AND CUPELLATION.

Scorification and cupellation include a combination of fusion, roasting, sublimation, and oxidation—the essential difference being that in the first case a slag is formed, and in the latter the foreign substances are volatilized or absorbed.

Scorification is essentially a process of oxidation. The test lead mixed with the ore melts, and the ore floats on the lead bath, where sulphur, arsenic, etc., are removed by volatilization and oxidation ; then the lead commences to oxidize and carry with it as a slag the gangue and oxides of the non-volatile metals. Gold, silver, and platinum remain with the lead button. The scorification is finished when the slag has covered the lead alloy.

Cupellation differs from scorification in that the oxide formed is volatilized or absorbed by the cupel as fast as produced. The process should only be used to remove small quantities of impurities with the lead. Gold, silver, and platinum remain. (See assay of gold and silver ores.)

### INQUARTATION AND PARTING.

Under this head come the separation of alloys and the treatment of the buttons from the gold and silver assay.

Inquartation is the process of alloying metals to give an

alloy from which one of the metals can be dissolved by an acid. The best proportion for gold and silver is 1 part of gold to about 2.5 parts of silver.

Parting is the separation of one or more metals from an alloy by means of acid : the term is usually applied to the separation of silver from a silver-gold alloy. Nitric acid of two strengths is used: first, sp. gr. 1.16, then, sp. gr. 1.26. Sulphuric acid, concentrated, is used in the mints and in special assays.

### WEIGHING BEADS AND BULLION.

This operation must be conducted with the greatest care, and the balance adjusted both before and after weighing. Before weighing, the bead or bullion should be well cleaned with a small brush. To weigh the buttons of the base metals the ore scales are sufficiently accurate; but for weighing silver and gold the bullion balance must be employed.

Take the weight from the vacant places in the box, note and check as you return the weights to their places in the box.

It is best to keep the bullion balance in a separate room from the laboratory, where it will be free from dust and fumes. It should also stand upon a firm shelf, to prevent shaking. In weighing a substance do not use the weights at random, but find the nearest single weight, and add the others in regular order, until the required combination is reached.

In duplicate assays the buttons should balance each other, or very nearly so.



To facilitate the weighing out of pure silver in the bullion assay, Mr. W. S. Ward of the U. S. Assay Office, in the city of New York, devised a series of standard discs, which run from fifty to five hundred milligrams. By taking one or more of these almost any desired weight can be obtained, thus saving labor and time. When obtaining a weight, the door of the balance should be kept closed, and the number of divisions marked by the needle observed, and also on which side of the centre-line they are. Each division counts  $\frac{1}{10}$  of a milligram on the second swing, and the total can either be added to or deducted from the weights in the pan, as the case may be; if the button is heaviest, add—if lightest, subtract. On the quantitative balance the rider indicates milligrams and fractions of the same; so that in obtaining the final weight after the pans are nearly balanced, the door can be closed and the rider adjusted by means of the rod from the right-hand side.

The modern bullion balances are provided with a rider arranged in the same way, which gives fractions (usually fiftieths) of a milligram.

Never lean on the balance shelf or leave the rider on the beam. The first may throw the balance out of adjustment; the second, cause error in the next weighing.

## TABULATING RESULTS AND REPORTING.

In making an essay each result should be noted as obtained, and nothing left to memory. Care should also be observed in arranging and reporting. To facilitate this, a series of blanks will be found in Part V, from which a choice can be made.

The report should be made as simple and comprehensive as possible, and written in terms which a business man can understand.

It should also indicate, in the case of gold and silver, the ounces Troy to the ton Avoirdupois, and the value in gold per ton of ore, gold being taken at \$20.67 per ounce Troy, silver variable. The value of silver by the old U. S. standard was \$1.29 per ounce; now, however, the market quotations per Troy ounce are taken and the value per ton calculated.

Base metals, such as lead, antimony, copper, etc., are reported in percentage. Gold and silver alloys are reported upon as to fineness, or the number of parts of each metal in a thousand parts of the alloy.



**PART II.**  
**DRY OR FIRE ASSAYS.**



## LEAD. *Symbol*—Pb.

SOURCES.—The principal ores of lead are :

|   |   |       |   |   |   |
|---|---|-------|---|---|---|
| Galena, sulphide ( $\text{PbS}$ ) containing 86.6 % of lead when pure   |   |       |   |   |   |
| Minium, oxide ( $\text{Pb}_3\text{O}_4$ ). . . . .  | “ | 90.   | “ | “ | “ |
| Cerussite, carbonate ( $\text{PbCO}_3$ ) “  | “ | 77.52 | “ | “ | “ |
| Anglesite, sulphate ( $\text{PbSO}_4$ ) “   | “ | 68.31 | “ | “ | “ |
| Pyromorphite, phosphate<br>and chloride ( $3\text{Pb}_3\text{P}_2\text{O}_8$<br>+ $\text{PbCl}_2$ ) . . . . . | “ | 76.36 | “ | “ | “ |

Lead also enters into the composition of many other minerals, such as Bournonite ( $3(\text{CuPb})\text{S}, \text{Sb}_2\text{S}_3$ ), Mimetite ( $3\text{Pb}_3(\text{AsO}_4)_2, \text{PbCl}_2$ ), Crocoite ( $\text{PbCrO}_4$ ), Stolzite ( $\text{PbWO}_4$ ), Wulfenite ( $\text{PbMoO}_4$ ), and Vanadinite ( $3\text{Pb}_3(\text{VO}_4)_2, \text{PbCl}_2$ ). They do not occur, however, in sufficient abundance to be classed as workable ores.

ASSAY.—The fire assay for lead, while inaccurate from a scientific standpoint, is very useful in determining the possible yield of an ore.

The following are the chief causes of inaccuracy in the lead assay :

- 1st. Loss of lead by oxidation and volatilization.
- 2d. Presence of substances which alloy with the lead—such as gold and silver in large quantities, iron, copper, zinc, antimony, bismuth, etc.—and so give an impure button.
- 3d. Presence of substances like arsenic which carry lead into the slag.

The assay of lead may be performed either in the crucible or muffle furnace, the methods of treating varying with the ores. The object of the assayer is in all cases to decompose the ore treated, and obtain a button of lead, slagging of the gangue and other impurities.

Methods applicable to sulphides, sulphates, etc.

| No 1.                 | No 2.            | No 3.                 |
|-----------------------|------------------|-----------------------|
| Ore ..... 10 gms.     | Ore .... 10 gms. | Ore ..... 10 gms.     |
| Soda ..... 30 “       | KCy .... 40 “    | Lead Flux 35 “        |
| Argol ..... 5 “       | Salt .... cover  | 4 Iron nails or wires |
| 4 Iron nails or wires |                  | Borax ..... cover     |
| Salt ..... cover      |                  |                       |

The ore and fluxes should be mixed thoroughly on glazed paper and then transferred to a small clay crucible, which should not be filled more than two-thirds full; then the salt for the cover is spread on the paper to collect any remaining particles of ore, and then placed on the top of the charge. The iron wires are bent double like hair-pins, and held together by a loop of wire which passes over the side of the crucible and is used to remove them quickly after the fusion is completed. The wires should reach to the bottom of the crucible.

Nails are used points down, held together by a wire. Smooth wire nails are the best.

Fresh fuel should be placed on the crucible fire, and the fire poked or shaken down until the bed of coals is solid; then the crucibles are put in, covered and surrounded with fresh fuel up to the top. If the charges are to be poured it is advisable to have the top well heated, so that it will not chill the slag in pouring.

The fire should not be very hot—just enough to ensure

complete fusion ; the time depends greatly on the ore and the fire, but is about half an hour. The assay is not finished until it has been in a state of quiet fusion for about five minutes, but the charge should not be left in the furnace longer than actually necessary.

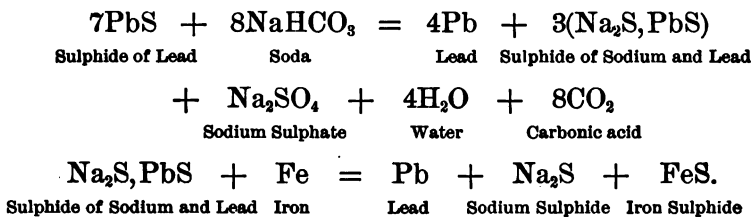
When finished, remove from the fire, take out wires or nails after shaking in the slag to detach particles of lead, tap the crucible gently on the floor to collect the lead in one button, cover, and allow to cool, or pour into moulds. When cold, detach slag and hammer the button into a cube.

If a muffle is used, half the preceding weights may be found more convenient.

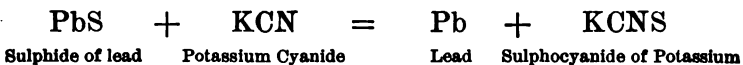
Several assays can be run in a muffle at once, but care in heating should be observed. The muffle should be at a bright cherry-red when the assays are introduced, and the heat raised until the cover fuses. This will take about twenty minutes, after which the muffle is made white hot for about ten minutes, when a perfectly fluid fusion is obtained.

If the ore has a basic gangue the addition of one or two grams of borax glass will help the fusion.

The reactions are as follows : Nos. 1 and 3 :

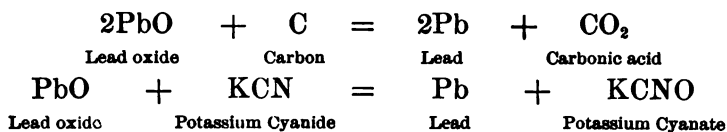


No. 2 :





Oxidized ores, carbonates, etc., can be assayed by preceding methods, omitting the nails or wires unless a little sulphide is suspected, when one loup of wire or a nail may be left in. These ores often require a higher heat and the addition of borax glass to flux a basic gangue. The reaction is a simple reduction.



Pyromorphite (lead phosphate) requires the addition of fluor spar ( $\text{CaF}_2$ ) or borax glass to prevent a pasty fusion from the sodium phosphate formed with the flux.

A good muffle method for assay is as follows: Charge ore 5 grams; flux, 20 grams of a mixture of sodium bicarbonate ( $\text{NaHCO}_3$ ) 2 parts, potassium carbonate ( $\text{K}_2\text{CO}_3$ ) 2 parts, borax glass ( $\text{Na}_2\text{B}_4\text{O}_7$ ) 1 part, argol 1 part; nails and salt as usual.

Lead slags can be assayed by fusing the pulverized slag as follows:

|                            |         |
|----------------------------|---------|
| Slag.....                  | 20 gms. |
| Argol .....                | 10 “    |
| Soda, Bicarb .....         | 40 “    |
| 2 Iron Nails, points down. |         |
| Salt.....                  | cover   |

The assay is conducted in the same way as in the treatment of ores. In very poor slags silver filings may be added as a collecting agent. The amount used must be carefully determined, and allowed for when the button is weighed.

REMARKS.—The following results on typical lead ores

give an idea of the accuracy of the assay and the relative merits of the different methods :

| Method.            | Galena.     | Galena and Pyrites. | Cerussite.  |
|--------------------|-------------|---------------------|-------------|
| Gravimetric.....   | 74.98       | 33.74               | 38.48       |
| No. 1, S. & A..... | 76.2 & 75.9 | 33.2 & 32.2         | 36.7 & 36.5 |
| No. 2, KCN.....    | 75. & 74.4  | 36.6 & 36.5         | 37.7 & 37.3 |
| Muffle method....  | 74.5 & 74.4 | 30.4 & 29.          | 37.3 & 36.9 |

### ANTIMONY. *Symbol*—Sb.

SOURCES.—The principal ore of antimony is the sulphide—called stibnite, or grey antimony ore ( $\text{Sb}_2\text{S}_3$ )—which contains, when pure, 71.80 per cent. of metal. Antimony is also found native combined with other metals, and also with oxygen.

ASSAY.—In the assay of antimony ore, the assayer may be required to determine one of two things :

*a.* The pure sulphide of antimony (antimonium crudum), which the ore may contain.

*b.* The metallic antimony (regulus of antimony), which the ore may yield.

*a.* Determination of the sulphide. As sulphide of antimony fuses at a low red heat, it is not changed in its character if the air is excluded, so that the following method may be adopted :

Charge the broken ore into a crucible the bottom of which is perforated, and which fits into a second crucible for about half its depth. Then cover, and lute the lid and the joint between the two crucibles with fire-clay and sand. The upper crucible only should be heated, and to effect this the lower can extend into the ash-pit of the furnace, being supported by an inverted crucible or a brick.

The sulphide of antimony will melt and collect in the

lower crucible, while the silicious and earthy matter remains in the upper.

*b.* Determination of metallic antimony.

1st. The ore is in the state of oxide.

|                                 |         |
|---------------------------------|---------|
| Ore .....                       | 10 gms. |
| Soda ( $\text{NaHCO}_3$ ) ..... | 25 "    |
| Argol .....                     | 5 "     |
| Salt .....                      | cover   |

This assay is conducted in the same manner as for lead, only the heat must be regulated with more care, and the assay taken from the fire as soon as finished ; the cover being left on the crucible while cooling.

The reaction is a simple reduction.

2d. The ore is a sulphide or oxide.

|                            |            |
|----------------------------|------------|
| Ore .....                  | 10 gms.    |
| Cyanide of Potassium ..... | 35 to 40 " |
| Salt .....                 | cover      |

The charge should be well mixed, the heat low, and the operation performed quickly ; observing the same precautions in cooling as in the preceding method.

This method is preferable for both oxides and sulphides.

**REMARKS.**—The result obtained in assaying for antimony cannot be accepted as the correct amount of metal in the ore ; it only represents the possible yield, as the button often contains some other metals, which have been reduced with the antimony in the ore, when the latter is not pure. It should, therefore, be tested for iron, etc., alloyed with the antimony. The button should be cleaned by washing, and not hammered, to detach the slag, as it is brittle.

When the ore contains much iron and silicious matter the second method does not give good results. Two assays of impure stibnite gave 44.5 and 44.2 per cent, two of oxide gave 73.2 and 73.4 per cent.

To separate from foreign metals, break the button and treat with concentrated nitric acid, which converts the antimony into antimonie acid, which is insoluble. Filter, wash, dry, and ignite in a porcelain crucible; the weight found, multiplied by 0.790067, gives the metallic antimony. Often it is not necessary to treat the buttons from the fire assay, as the loss by volatilization more than counterbalances the impurities in the button.

For some impure ores a very large charge of cyanide (say 50-60 gms.) and a quick fire has been found to give good results.

### BISMUTH. *Symbol*—Bi.

SOURCES.—This metal is found principally in the metallic state, but it also occurs in combination with sulphur, oxygen, and tellurium, associated with lead and silver. Bismuth, like lead, possesses the property of causing the absorption of the metallic oxides in cupellation, and may be used in place of the latter, but is not recommended.

ASSAY.—In the assay for bismuth three cases may occur.

- a. The sample contains native bismuth.
- b. The sample is composed of bismuth with other substances, or bismuth residue.
- c. The sample is an alloy.

*a.* Determine as in the assay for "antimonium crudum," the bismuth being collected in the same way.

*b.* Pulverize finely and charge:

|                            |         |
|----------------------------|---------|
| Ore.....                   | 10 gms. |
| Cyanide of Potassium... .. | 40 "    |
| Salt.....                  | cover   |

Some assayers replace part of the cyanide of potassium with a mixture of carbonate of soda and potash and for poor ores use 20 gms. instead of 10 gms. of ore.

Fuse in a moderate fire in the same manner as for antimony. The resulting button must be tested for other metals, and if any be present, treated as an alloy.

*c.* Determine by the wet assay. (See Part III.)

REMARKS.—Bismuth melts at 268° C., and is volatile at a higher temperature.

The assay for bismuth may also be made by fusing the pulverized and sintered ore (prepared by heating alone in a closed crucible) with a known weight (five to ten gms.) of fine silver, black flux, and three to five grams of iron wire, covering with salt. The button can afterward be treated as an alloy. Plattner's Manual of Blowpipe Analysis, page 459.

A button of bismuth should not be hammered, as it is brittle.

For the accurate determination of bismuth wet methods must be used.

TIN. *Symbol*—Sn.

SOURCES.—The most abundant ore is cassiterite, binocide ( $\text{SnO}_2$ ), = 78.67 per cent. when pure. It is found in veins and in the washings from the same under the name of stream tin; sometimes it is associated with tungsten, tantalum, or molybdenum. Tin also occurs as a sulphide in stannite, tin pyrites  $\left\{ \begin{smallmatrix} \text{Cu}_2\text{S} \\ \text{FeS} \end{smallmatrix} \right\} \text{SnS}_2$ , and rarely in the native state.

In the United States, high grade tin ore has been found only in small quantities, most of the ore not being rich enough to pay for working.

The treatment of tin ores in the laboratory is a matter of some difficulty for several reasons:

1st. The ore is often associated with a gangue, the constituents of which either form salts with the oxide of tin or alloy with the reduced metal from the same.

2d. The majority of the basic fluxes at the disposal of the assayer combine with the tin and oxygen which may be present, forming stannates which go into the slag.

3d. Acid fluxes, especially silica, form compounds with the oxide of tin, and carry it into the slag. The influence of silica can be seen by the following table, given by Mitchell. The last line shows the yield of metal:

|                  |       |       |       |       |            |
|------------------|-------|-------|-------|-------|------------|
| Ore . . . . .    | 10.00 | 10.00 | 10.00 | 10.00 | 10.00 gms. |
| Silica . . . . . | 2.50  | 6.60  | 10.00 | 15.00 | 30.00 “    |
| Tin . . . . .    | 52%   | 43%   | 28%   | 10%   | none       |

The fusion was made in each case with the same quantity of black flux.

4th. Binoxide of tin is extremely difficult to fuse; it is insoluble, even in concentrated acids, and although it is reducible by ignition with hydrogen, charcoal, etc., there is always danger of loss if the temperature be too high, as tin boils at a white heat, air being excluded to prevent oxidation.

ASSAY.—This will generally include a preliminary treatment, unless the ore is very pure and free from gangue, and the assay proper.

*Preliminary Treatment.*—The sample of ore taken for assay should contain a high percentage of tin, and should be as free as possible from impurities.

To remove the gangue take a weighed quantity—from 50 to 200 grams—of the ore, ground to 60 mesh, and pan down under water till only the heavy cassiterite and associated minerals remain, dry, weigh, and either use all or a part of the concentrates for assay. If all of the concentrates are used, the weighing need only be done approximately, so as to get the proper quantity of flux, etc., as the percentage of tin is reported from the weight taken before panning.

If the concentrates contain sulphur or arsenic, they should be roasted, first alone, then with a little carbonate of ammonia to decompose sulphates. If there is much oxide of iron associated with the cassiterite, transfer the residue from the roasting-pan to a casserole, and boil with strong hydrochloric acid till all the iron has dissolved, dilute, decant, wash, dry, and use the residue for assay.

*Assay Proper.*—The assay proper may be divided as follows:

a. Methods giving tin buttons.

b. Methods giving alloys from which the tin may be calculated.

c. Methods giving an alloy to be treated subsequently in the wet way.

a. *German Method (Modified)*.—Ten grams of the concentrated ore are mixed with 2 grams of powdered anthracite and put in the bottom of a No. 5 Hessian crucible; on this is placed a mixture of 30 grams black flux substitute and 1 gram borax glass, then a cover of salt, and finally several small pieces of charcoal. The crucible is heated in a hot coke fire for about one hour. The right amount of heat varies somewhat with the ore; for Mexican ores the heat should be between red and white. The buttons should be bright, should not have a burned appearance, and should be easily cut. Care must be taken to collect any small tin buttons which may be in the slag.

This assay can be made in a muffle, using half the preceding charge.

The theory of the assay is that the charcoal mixed with the ore reduces the tin, at least in part, before the black flux substitute fuses; then this melts, completes the reduction, slags off the gangue, and collects the button. The charcoal on top is only a precaution against oxidation from outside influences.

*Cyanide Method*.—Put 5 grams of potassium cyanide in the bottom of an unlined Hessian crucible, then 10 grams of the concentrated ore mixed with 30 grams of cyanide, then a layer of 5 grams of cyanide and a cover of salt. Fuse at a good red heat for thirty minutes. The regulation of the heat is very important in this method; too hot a fire reduces iron which gives a hard dull button.

This assay can be run in a muffle, using half the charge, or can be modified as follows:



Ore, 2 to 5 grams ; KCy, 10 to 15 grams ; mix, place in a porcelain capsule and fuse for ten minutes in the muffle. When cool, place in a beaker of water to dissolve the slag, which is largely cyanide and cyanate of potassium, collect, and weigh the particles of tin.

*b. Winkler's Method (Modified).*—Put 5 grams of potassium cyanide in the bottom of a No. 5 Hessian crucible, then 10 grams of concentrated ore mixed with 10 grams of oxide of copper ( $\text{CuO}$ ) and 30 grams of cyanide, then a layer of 5 grams of cyanide, then a cover of salt. Heat in a moderately hot fire for one hour.

With every set of assays run a blank, using above charge omitting the tin ore, to determine the copper actually reduced. Subtract the weight of the copper reduced from that of each of the bronze buttons, and the result multiplied by 10 gives the percentage of tin by this method.

*c.* Take such quantities of ore and oxide of copper as to give a button by the preceding method of about three grams, of which at least two grams will be copper. Treat the button with nitric acid until all the copper has dissolved, dilute, and boil to ensure complete precipitation of the hydrated oxide of tin. Filter, wash, ignite, and weigh the  $\text{SnO}_2$ . Calculate to tin.

REMARKS.—A tin ore from Durango, Mexico, gave the following results: \*

|                                 |       |           |
|---------------------------------|-------|-----------|
| Analysis Method of Rose . . . . | 65.62 | per cent. |
| (a) German Method (Average).    | 63.92 | “ “       |
| (a) Cyanide Method “            | 65.19 | “ “       |
| (b) Winkler's Method “          | 63.95 | “ “       |
| (c) Nitric Acid Method “        | 63.92 | “ “       |

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\* Miller, S. of M. Quarterly, No. 4, Vol. XIII.

Tin assays should be run in duplicate, and the average, not the highest, result reported. Duplicates should agree within 2 per cent. on a rich ore.

### IRON. *Symbol*—Fe.

SOURCES.—The following is a list of the principal ores of iron :

|  |                                   |   |   |   |   |
|--|-----------------------------------|---|---|---|---|
| Magnetic iron ore, magnetite, oxide, ( $\text{Fe}_3\text{O}_4$ )..                             | Cont'g 72.41 % of iron when pure. |   |   |   |   |
| Red hematite or specular iron, oxide, ( $\text{Fe}_2\text{O}_3$ )...                           | " 70.00                           | " | " | " | " |
| Brown hematite or limonite, oxide, ( $2\text{Fe}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$ )..... | " 59.92                           | " | " | " | " |
| Spathic iron ore, siderite, carbonate, ( $\text{FeCO}_3$ )...                                  | " 48.22                           | " | " | " | " |
| Ilmenite(mennaconite), titaniferous ore ( $\text{FeTiO}_3 + n\text{Fe}_2\text{O}_3$ ).....     | " 36.82                           | " | " | " | " |
| Franklinite ((Fe,Zn,Mn) ( $\text{Fe}_2\text{Mn}_2\text{O}_4$ )).....                           | " 48.00                           | " | " | " | " |
| Chromite ( $\text{FeCr}_2\text{O}_4$ ). Also sulphides, as pyrite, pyrrhotite, etc.            |                                   |   |   |   |   |

ASSAY.—It is required in the assay for iron not only to reduce the oxide to cast iron, collect the latter in a button, and to form a fusible slag that will not retain any of the iron in combination or in pellets, but also to use such fluxes and so adjust them, that the results will indicate the character of the ore, quality of iron it will yield on smelting, etc. The oxide is reduced by carbon, and we employ for this purpose crucibles lined with brasque, which is a

mixture of four parts finely pulverized charcoal to one part of molasses. (To prepare this see page 22.)

The lining also serves as a support for the crucible, which under the high heat is apt to soften.

In making up the charge we may have, (1) ores of unknown composition, and (2) ores previously analyzed. The assay in both cases gives a clue to the nature of the slag, the iron that may be obtained from the ore, and the character and proportion of the fluxes to be added in the blast-furnace. In the first case we obtain additional information as to the approximate percentage of iron.

#### 1. Ores of unknown composition.

In the assay of an ore the composition of which is unknown we make several preliminary assays, and if satisfactory results are obtained we make another assay with a charge modified according to the indications of the best preliminary assay.

#### Preliminary assay charges :

|             | 1.   | 2.   | 3.   | 4.             |
|-------------|------|------|------|----------------|
| Silica..... | 2.5  | 1.0  | 4.0  | 2.5 to 0. gms. |
| Lime.....   | 2.5  | 4.0  | 1.0  | 2.5 to 3. “    |
| Ore.....    | 10.0 | 10.0 | 10.0 | 10.0 “         |

1, is employed for the purer ores containing very little gangue ; 2, for ores containing silica ; 3, for ores containing the carbonates of lime or magnesia, protoxide of manganese, etc., calcareous hematites and spathic iron ; 4, for ores containing silica and alumina, clay ironstones, black-band, etc.

The principle involved is that of furnishing a base for an acid, and *vice versa*. The charge, therefore, depends upon the acid or basic nature of the gangue of the ore.

Ores containing titanium require the addition of fluor-spar to the charge in quantity varying from 0.5 to 10 gms.

## 2. Ores previously analyzed.

Good results are obtained from a charge proportioned to yield a slag corresponding to a blast-furnace cinder, having the composition  $2(2\text{RO}, \text{SiO}_2) + 2\text{R}_2\text{O}_3, 3\text{SiO}_2$ , called Percy's slag.

$\text{R}_2\text{O}_3$  represents alumina, and RO lime, magnesia, and other bases. Its approximate percentage composition is as follows :

|                                       |    |            |                       |   |
|---------------------------------------|----|------------|-----------------------|---|
| Silica.....                           | 38 | } or about | $2\frac{1}{2}$ parts. |   |
| $\text{R}_2\text{O}_3$ (alumina)..... | 15 |            | 1                     | " |
| RO (lime, magnesia, etc.)..           | 47 |            | 3                     | " |

The method of charging can best be shown by an example:

| The Ore Contains     | Per Cent. | 10 gms. of Ore Contain | Slag Required. | Difference to be Added. |
|----------------------|-----------|------------------------|----------------|-------------------------|
| Silica.....          | 1.65      | 0.165 gms.             | 2.50 gms.      | 2.335 gms.              |
| Alumina.....         | 1.94      | 0.194 "                | 1.00 "         | 0.806 "                 |
| Lime, magnesia, etc. | 4.51      | 0.451 "                | 3.00 "         | 2.549 "                 |

The alumina is added in the form of kaolin or fire-clay, which contains nearly equal parts of alumina and silica. Allow in adding silica for that introduced in the kaolin.

Sometimes the ore contains an excess of one of the ingredients of the slag, or the silica introduced with the kaolin may, when added to that already present, increase the quantity beyond what is required. In either case make up a new slag with the excess, retaining the same proportion between the silica, alumina, and lime, viz. : silica,  $2\frac{1}{2}$ ; alumina, 1; lime, 3. For examples, see Part V.

The charge should be thoroughly mixed, placed in the crucible, the conical cavity closed with a piece of charcoal,

and the whole top of the crucible covered with a luting of fire-clay. The latter is a mixture of clay with one fourth to one third part of fine sand made plastic with borax-water.

Four crucibles are introduced in the furnace luted to fire-bricks, and a low fire kindled around them. The fuel is added gradually until it is above the tops of the crucibles; the fire is maintained at its maximum temperature for two and one half to three and one half hours. Ores containing much titanium require four hours, while carbonates containing manganese fuse well in two and one half hours, or even less time. When the fire has burned out, the bricks and crucibles are removed in one mass, allowed to cool, the crucibles detached and their exteriors broken with a hammer; on inverting and tapping the charcoal lining the slag and button of cast iron will fall into the hand. If these adhere together, a slight tap serves to separate them, but before separation they should be carefully cleansed and weighed; the slag may then be broken, and any particles of iron removed with a magnet, and weighed with the button.

Titanium and manganese enter the slag almost completely. Duplicate assays should not differ more than 0.3 to 0.4 of one per cent. The slag ought to be well fused and free from iron. A good button is well formed and easily detached from the slag.

If the metal be of good quality, the button will flatten slightly before breaking. It ought to be gray or grayish white, and the grain fine.

A button of bad iron breaks readily without changing form.

Transparent slags of a greenish tint indicate excess of silica. A rough, stony slag, or one crystalline in struc-

ture and dull in lustre, indicates an excess of base. If the product is only fritted and contains the reduced iron interspersed as a fine gray powder, silica and alumina are deficient in the flux, lime and magnesia being in excess.

Magnesia is one of the most refractory bases found in iron ores, and when present in quantity requires an addition of both silica and lime.

Manganese gives an amethystine tint to the slag, or if in excess a yellow, green, or brown color.

Titanium produces a resinous, black, scoriaceous slag, curiously wrinkled on the outside, and covered with metallic pellicles of nitro-cyanide of titanium with its characteristic copper color; sometimes the slag is vitreous and of a bluish tint.

Chromium gives a dark resinous slag surrounded with a thin metallic coating.

Phosphorus gives a hard, brittle, white metal, called cold-short iron.

Sulphur gives a strong, reticulated, mottled structure, and red-short iron.

Manganese gives a button smooth on the exterior, hard and non-graphitic; it presents a white crystalline fracture.

Titanium.—The button is smooth on the outside, has a deep gray fracture, and adheres strongly to the slag: it is sometimes covered with nitro-cyanide of titanium.

Chromium.—The button is smooth, well fused, with a brilliant crystalline fracture, and tin-white color; sometimes it is white and only half fused, or it may even form a spongy looking mass of a clear gray color, according to the quantity of chromium contained in the iron.

REMARKS.—A number of typical ores gave on assay :

| Ore.            | Iron by Analysis. | By Fire Assay.          |      |                |
|-----------------|-------------------|-------------------------|------|----------------|
| Magnetite. .... | 68.35 per cent.   | 69.6                    | 71.2 | 71.3 per cent. |
| Hematite. ....  | 44.50 “           | 44.6                    | 46.0 | 48.6 “         |
| Limonite. ....  | 44.20 “           | 44.3                    | 44.6 | 45.2 “         |
| Magnetite ..... |                   | 64.0 and 64.5 per cent. |      |                |
| Hematite .....  |                   | 39.0 “ 38.5 “           |      |                |
| Limonite .....  |                   | 34.0 “ 34.0 “           |      |                |

Assays of magnetite containing titanitic acid gave 72.5 and 73 per cent.

Other slags besides the one given might be used, as Bodemann's, ( $\text{CaO}, \text{SiO}_2 + \text{Al}_2\text{O}_3, 3\text{SiO}_2$ ) which is: silica, 56; lime, 30; alumina, 14 per cent.; an addition of a little borax and fluor-spar makes this slag more fluid.

It is very important from an economic standpoint to have a minimum amount of slag, and in an ore containing an excess of silica, if a new charge of slag is made up to provide for the excess this should be kept in mind, to prevent waste of fuel and flux. In Percy's slag  $\text{SiO}_2$  2.5 :  $\text{Al}_2\text{O}_3$  1 :  $\text{CaO}$  3, besides being the proper ratio of acid to bases, is the weight in grams of slag required for ten grams of ore.

The following example illustrates the methods of calculating the minimum correct slag when silica is in excess :

| Suppose an ore contains |              | In 10 Grams. | Required.  | To Add.    |
|-------------------------|--------------|--------------|------------|------------|
| $\text{SiO}_2$          | 22 per cent. | 2.2 grams.   | 2.5 grams. | 0.3 grams. |
| $\text{Al}_2\text{O}_3$ | 4 “ “        | 0.4 “        | 1.0 “      | 0.6 “      |
| $\text{CaO}$            | 15 “ “       | 1.5 “        | 3.0 “      | 1.5 “      |

We may add silica or lime direct, but as pure alumina is difficult to obtain, the deficiency of alumina is made up

by adding kaolin, which is half alumina and half silica. Make the alumina right by adding kaolin 1.2 grams. The excess of silica is now 0.3 of a gram. Make up a new amount of slag for this excess as follows: Let  $x$  = weight of kaolin to be added; then  $\frac{x}{2}$  equals alumina, and  $\frac{x}{2}$  equals silica,  $\frac{x}{2} + 0.3 : \frac{x}{2} :: 2\frac{1}{2} : 1$ , or the silica added + the silica present is to the alumina added as  $2\frac{1}{2}$  is to 1. Solving gives  $x = 0.4$ .

Lime is always three times the alumina, hence lime to add =  $1.5 + 3\left(\frac{x}{2}\right) = 2.1$  grams; 1.5 grams is the original deficiency in lime, 0.6 of a gram is the lime needed by the excess of silica.

Proof:  $\text{SiO}_2 \quad 2.2 + 0.6 + 0.2 = 3.0 \div 1.2 = 2.5$ .

$\text{Al}_2\text{O}_3 \quad 0.4 + 0.6 + 0.2 = 1.2 \div 1.2 = 1.0$ .

$\text{CaO} \quad 1.5 + 1.5 + 0.6 = 3.6 \div 1.2 = 3.0$ .

This example may also be done as follows:

Take the number of grams of  $\text{Al}_2\text{O}_3$  in 10 grams of ore as unity.

|                            | In 10 Grams. | Required. | Correct Proportion. | Excess. |
|----------------------------|--------------|-----------|---------------------|---------|
| $\text{SiO}_2$ 22%         | 2.2 grams.   | 2.5 grams | 1.0                 | 1.2     |
| $\text{Al}_2\text{O}_3$ 4" | 0.4 "        | 1.0 "     | 0.4                 |         |
| $\text{CaO}$ 15"           | 1.5 "        | 3.0 "     | 1.2                 | 0.3     |

Make up slag for the excess:

$$\frac{x}{2} + 1.2 : \frac{x}{2} :: 5 : 2; x = 1.6.$$

Lime to add:  $1.6 \times \left(\frac{3}{2}\right) - 0.3 = 2.1$ .

The result is the same by both methods. Kaolin to add, 1.6 grams; lime, 2.1 grams.



COPPER. *Symbol*—Cu.

SOURCES.—The substances containing copper may be divided into three classes :

1st. Pure or oxidized ores.

2d. Impure ores, or compounds of copper and other metals with sulphur, arsenic, antimony, etc.

3d. Native copper and alloys.

The most abundant ores of copper are :

|   |                   |                     |
|---|-------------------|---------------------|
| Native copper and its silver alloy....  | Containing 100.0% | } Copper when pure. |
| Cuprite, red oxide ( $\text{Cu}_2\text{O}$ ).....                                     | “ 88.8 “          |                     |
| Malachite, carbonate and hydrate<br>( $\text{CuCO}_3 + \text{CuH}_2\text{O}_2$ )..... | “ 57.4 “          |                     |
| Azurite, carbonate and hydrate<br>( $2\text{CuCO}_3 + \text{CuH}_2\text{O}_2$ ).....  | “ 55.3 “          |                     |
| Chalcocite, copper glance ( $\text{Cu}_2\text{S}$ )...                                | “ 79.8 “          |                     |
| Chalcopyrite, copper pyrites (Cu<br>FeS <sub>2</sub> ).....                           | “ 55.5 “          |                     |
| Bornite, purple copper ( $\text{FeCu}_3\text{S}_3$ )...                               | “ 34.5 “          |                     |
| Enargite ( $\text{Cu}_3\text{AsS}_4$ ).....   | “ 48.3 “          |                     |

Compounds with arsenic, antimony, lead, mercury, etc., the chloride, atacamite, and the silicate, chrysocolla.

ASSAY.—Copper can best be determined in the dry way by roasting the ore with carbonate of ammonia and then fusing with arsenic and slagging off the other arsenides combined with it. This method is exact, but requires practice. It serves to determine, besides the copper, lead, bismuth, cobalt, and nickel. The crucible used for the fusion is shown in Fig. 36. The assay can be



FIG. 36. conducted in the muffle furnace, and involves the following operations :

- a. Roasting with ammonium carbonate.
- b. Treatment of the roasted ore with metallic arsenic.
- c. Fusion with fluxes to collect the arsenides in a button and to separate lead and bismuth.
- d. Separation of the arsenide of iron, etc., from the arsenides of nickel, cobalt, and copper.
- e. Separation of the arsenide of cobalt.
- f. Fusion with gold and separation of arsenide of nickel.

For details of the assay see method for nickel and cobalt, pages 84 to 87; also Watts' Dictionary of Chemistry, Vol. II., page 63.

The dry assay for copper has been almost entirely superseded by the electrolytic and volumetric methods. These are fully described in Part III. Should the assayer not have the apparatus required for the wet assay, the following method will afford an approximate idea of the percentage of copper in a rich ore or matte.

*Charge for Copper Matte.*—Matte 10 grams; roast alone, then with ammonium carbonate, and then with charcoal; mix with potassium cyanide, 30 grams; borax glass, 3 grams; and place in a No. 5 Hessian crucible; cover with 10 grams of potassium cyanide, then with salt; fuse in a hot fire for about half an hour.

REMARKS.—A matte, free from antimony and zinc, containing 54 per cent. of copper gave by the cyanide method buttons weighing 5.33, 5.36, and 5.34 grams. Such satisfactory results cannot be obtained with ores or mattes containing other metals which will be reduced and alloy with the copper. For ores, 2 to 4 grams of oxide of iron, hematite, should be added to the charge to prevent the loss of copper in the slag. If the resulting button is

evidently impure, it can be refined by fusing in a scorifier in the muffle with a little pure lead, 1 to 2 grams, and about the same amount of borax glass; when the copper is nearly refined, it brightens and shows a peculiar green color. There is generally considerable loss of copper in the slag.

### NICKEL AND COBALT. *Symbols*—Ni and Co.

SOURCES.—These two metals are generally found associated, and their treatment will be described under the same head.

The principal ores of nickel are niccolite, copper nickel ( $\text{NiAs}$ ), = 43 per cent. nickel when pure; Millerite, sulphide ( $\text{NiS}$ ), = 64.4 per cent. nickel when pure.

The principal ores of cobalt are smaltite, tin-white cobalt ( $(\text{Co}, \text{Fe}, \text{Ni})\text{As}_2$ ), = 9 to 33 per cent. cobalt when pure; cobaltite ( $\text{CoS}_2 + \text{CoAs}_2$ ), = 35.5 per cent. cobalt when pure.

Both nickel and cobalt occur in many other minerals, chiefly in combination with sulphur or arsenic, and associated with iron, copper, lead, etc.

We have also an artificial product called "spiess," which is an arsenide of cobalt, nickel, and iron, obtained in the smelting of ores which contain nickel and cobalt, and in the manufacture of smalt (cobalt glass).

ASSAY.—Cobalt and nickel are difficult to fuse, and are therefore determined in combination with arsenic. Weigh out from two to five grams according to the purity of the ore, roast thoroughly in the muffle, using a clay roasting-dish, and mixing with six to ten grams of fine charcoal toward the end of the operation. When the sample has

ceased to evolve fumes, mix thoroughly with one to five grams of ammonium carbonate, and heat. The resulting oxides are then converted into arsenides by moistening and rubbing in a mortar with one to five grams of metallic arsenic, and heating the mixture in a small clay crucible (Fig. 36), which will stand in a muffle. Keep at a dull red until the fumes of arsenic have ceased ; then the crucible is removed, and about thirty grams of black flux, or its substitute, and one or two grams of borax glass are added, with a covering of salt. Do not mix the flux, but place it over the mass in the crucible ; after which heat in a good fire, raising the temperature gradually, until the contents of the crucible are in a quiet state of fusion. Make the heat strong toward the end of the operation, but be careful not to let the charge boil over. Cool and break the crucible, remove and weigh the button ( $\alpha$ ) of arsenides of cobalt, nickel, iron, copper, etc. The rest of the process consists of scorifying the button, first to remove the iron, and afterwards to separate the nickel and cobalt. This can be done in a shallow dish, or on a piece of clay crucible about two inches long by one inch wide and slightly concave, in the cupel muffle, which should be very hot and contain a piece of glowing charcoal in front. Place on the dish the button of arsenides from the fusion and cover it with borax glass. Introduce it into the muffle and close the latter until the button and borax are fused, then allow air to enter. The arsenide of iron will oxidize first and go into the slag, and the surface of the button will become bright, then the dish should be removed immediately, placed upon the surface of a basin of water until the button solidifies, and then immersed ; or the button can be removed with a pair of pincers while the

slag is fluid. If the slag shows a slight blue color, the iron is entirely removed, and the button may be cleaned and weighed (*b*).

If the desired purity from iron is not obtained by one scorification, repeat the operation, weighing the button each time. Should the button become bright immediately, showing that little or no iron was contained, take the previous weight. The button will consist of the arsenides of nickel, cobalt, and copper ( $\text{Ni}_3\text{As}_2$ ,  $\text{Co}_3\text{As}_2$ ,  $\text{Cu}_3\text{As}_2$ ).

Next slag off the cobalt in the same manner as the iron. This operation must be continued until an apple-green film forms, which will float about on the surface of the button, best seen by partially cooling the assay. Weigh the button (*c*).

If copper is present, add 100 to 500 milligrams of gold (weighed), and then proceed to slag off the nickel with the addition of a little salt of phosphorus, conducting the operation as before until the button shows the bluish-green color peculiar to an alloy of gold and copper when melted. Weigh the button of gold and copper (*d*).

To determine the weight of copper subtract the weight of gold added from the button (*d*). The difference will be the metallic copper. To determine the weight of nickel multiply the weight of the copper by 1.3935; this will give the arsenide of copper ( $\text{Cu}_3\text{As}_2$ ), which subtracted from the weight (*c*) will give the arsenide of nickel. This multiplied by 0.60986 = the nickel. To determine the cobalt subtract the weight (*c*) from (*b*) and multiply by 0.61083.

The results thus obtained divided by the weight of ore taken for assay and multiplied by 100, gives the percentage in each case.

REMARKS.—Results compare well with the battery process. The following is the composition of the arsenides :

| $\text{Cu}_6\text{As}_2$ . | $\text{Ni}_4\text{As}_2$ , | $\text{Co}_4\text{As}_2$ , |
|----------------------------|----------------------------|----------------------------|
| As = 28.24 per ct.         | As = 39.01 per ct.         | As = 38.92 per ct.         |
| Cu = 71.76 “               | Ni = 60.99 “               | Co = 61.08 “               |

When the ore treated contains bismuth and lead in any quantity, these metals can be separated during the fusion with black flux by adding one gram of iron wire, and one to three grams of pure silver, accurately weighed. After fusion the lead and bismuth will be found alloyed with the silver, and can be detached without trouble from the arsenides. By deducting the silver the lead and bismuth may be determined. When the substance treated is poor in nickel and cobalt, it is well to add some collecting agent in the fusion. Arsenide of iron is the best for this purpose ; it may be prepared by fusing iron filings with metallic arsenic in a crucible and powdering.

### MERCURY. *Symbol*—Hg.

SOURCES.—The principal ore of mercury is cinnabar, sulphide ( $\text{HgS}$ ), = 86.27 when pure. It also occurs in the metallic state, alone and amalgamated with silver, gold, etc., and is sometimes found combined with chlorine, as horn mercury ( $\text{Hg}_2\text{Cl}_2$ ).

Some varieties of tetrahedrite (gray copper) contain from 0.5 to 17 per cent. of mercury.

ASSAY.—The determination of mercury is made by distillation.

(1) *Ores, Usually Sulphides*.—100 to 500 grams of the ore according to the richness, is mixed with a desulphuriz-

ing and a reducing agent, and charged in a retort. The following reagents may be used:

Black flux, weight equal to weight of ore.

Black flux substitute, 150 per cent. of weight of ore.

Iron filings, 50 per cent., and charcoal, 30 per cent.

Lime, 30 per cent., and charcoal, 30 per cent.

The retort may be of iron, glass, or clay (glazed) with a bent neck, the end of which is placed under water in a glass vessel. It is better also to wrap the neck of the retort with a damp cloth. The retort may be heated over a small charcoal furnace, or in any way by which the heat can be applied slowly, and the whole body of the retort heated to prevent condensation of the mercury on the top. When after heating some time no more mercury comes over, the end of the neck should be lifted out of the water to prevent its being drawn over into the retort. The latter is allowed to cool slowly, and all adhering particles of the metal are brushed with a feather into the glass receiver, where they can be collected by boiling the water for a moment. The water is then decanted, and the mercury dried at the ordinary temperature or with blotting-paper and weighed on glass.

(2) *Metallic Mercury and Amalgams.*—Distill without the addition of any decomposing agent, otherwise conducting the operation as above. The heat used need not be so high, mercury being very volatile. For the treatment of amalgam small iron crucibles, with an escape-tube for the mercury, can be purchased of almost any apparatus-dealer.

REMARKS.—For all distillations the retort must be tight. For this reason glass or iron retorts are the best. Earthen retorts should be glazed. The operation should

be conducted under a hood, care being taken not to inhale any of the fumes.

The wet method is preferable for mercury ores.

See Mitchell, page 453 ; Goodyear's translation of Bode-mann and Kerl, page 207 ; and Kerl's Assayers' Manual (Garrison), page 236.

The mercury can be conveniently removed from small quantities of amalgam to be assayed for gold and silver by placing in a scorifier, and inverting over it a second scorifier of the same size, through the bottom of which a small hole has been bored ; the two scorifiers are luted together and heated gently in the muffle until the mercury has been expelled. The top scorifier is then removed, test-lead, etc., added to the residue, and the assay completed as usual for the determination of the gold and silver.

#### GOLD AND SILVER. *Symbols*—Au and Ag.

SOURCES.—All substances containing gold and silver may, for the purpose of the assayer, be divided into two classes:

Class 1. Minerals or ores, including incidental industrial products.

Class 2. Metallic gold and silver and alloys, native or artificial. (See Assay of Gold and Silver Bullion.)

*Ores*.—The principal ores of gold are native gold, usually in a quartz gangue or associated with pyrite (iron pyrites) ( $\text{FeS}_2$ ), chalcopyrite (copper pyrites) ( $\text{CuFeS}_2$ ), arsenopyrite (mispickel) ( $\text{FeS}_2, \text{FeAs}_2$ ), sphalerite (blende) ( $\text{ZnS}$ ), galena ( $\text{PbS}$ ), tetrahedrite (gray copper) etc., also



compounds of gold, such as sylvanite, hessite, and other tellurides.

The more important silver ores are native silver, argentite or silver glance ( $\text{Ag}_2\text{S}$ ), stephanite ( $5\text{Ag}_2\text{S}, \text{Sb}_2\text{S}_3$ ), pyrrargyrite (dark ruby silver) ( $3\text{Ag}_2\text{S}, \text{Sb}_2\text{S}_3$ ), proustite (light ruby silver) ( $3\text{Ag}_2\text{S}, \text{As}_2\text{S}_3$ ), cerargyrite (horn silver) ( $\text{AgCl}$ ). Silver is also found associated with stibnite and with the same minerals as gold.

For a more complete list of gold and silver minerals see Part V.

ASSAY OF ORES.—Assays of gold and silver ores are made in almost the same manner, so that a general description will answer for both. They embrace the following steps :

- 1st. Preparation of the sample.
- 2d. Collection of the gold and silver in a lead button.
- 3d. Scorification of the lead button, if necessary.
- 4th. Cupellation of the lead button.
- 5th. Weighing the gold and silver bead.
- 6th. Inquartation and parting (separation of the gold).
- 7th. Weighing the gold.
- 8th. Calculating results and reporting.

*Preparation of the Sample.*—Great care should be taken in grinding and sampling gold and silver ores. All apparatus must be clean and free from the ore previously pulverized. The ore should be ground to from 80 to 100 mesh unless it contains scales of precious metal; in this case see page 104, also under Sampling, Part I.

*Collection of the Gold and Silver in a Lead Button.*—This is effected in a crucible or scorifier, hence two methods of assay: (a) Crucible. (b) Scorification.

(a) *Crucible Assay.*—This is best adapted for all gold

ores with the exception of very rich tellurides, which may be run by scorification assay, for low-grade silver ores, and for pure rich silver ores having a basic gangue, or "carbonate" ores, and for chloride ores. One great merit in the crucible assay is the large amount of ore taken, which affords a much better average than can be obtained by scorification, and thus ensures accurate results, especially for gold, from ores and tailings which would show nothing or only a trace by scorification.

The theory of the crucible assay is that enough oxide of lead shall be reduced throughout the charge to collect all of the gold and silver, that the fluxes shall combine with the gangue of the ore so as to give an easily fusible slag, usually a double silicate, and that the oxidizing or reducing agents added will regulate the reduction and give a button of the proper size, viz., about 18 grams.

The charge of ore, fluxes, etc., cannot be given in an absolute form, as all the ingredients except perhaps the salt cover, are variable.

The amount of ore taken varies from  $\frac{1}{2}$  to 4 A.T., depending on its richness. The litharge varies from 1 A.T. to 6 A.T. The quantity of soda is usually the same as of ore.

Silica varies from 0 to 3 A.T., depending on the character of the gangue; basic gangues, such as limestone, dolomite, barite, fluor-spar, siderite, clays, and metallic oxides, require much silica, while acid gangues, like quartz, sandstone, granite, feldspar, and some clays, require but little or none. It should be remembered, therefore, that a basic ore will require an acid flux, and an acid ore a basic flux.

The oxidizing and reducing agents depend on the presence or absence of arsenic, antimony, zinc, and sulphur,

the common reducing impurities, and of oxides of manganese or iron which oxidize.

In the case of a pure ore, such as native gold in quartz, the amount of reducing agent to be added should be that which will reduce about 18 grams of lead, say 1.5 to 2 grams of argol, or 0.75 of a gram of charcoal.

*Preliminary Assay.*—If the ore contains sulphides, etc., it is often necessary to make a preliminary assay to determine its reducing power, and obtain data to regulate the size of the button.

Charge: ore, 5 grams; litharge, 80 grams; soda, 20 grams; borax glass, 5 grams; mix as usual, place in a small sand crucible, and cover with salt. The fire should be bright and effect the fusion in about 15 minutes. When the fusion is quiet, withdraw, tap, cool, break, hammer, and weigh the button.

Four cases may arise:

1st. No lead reduced. This shows that the ore either has no reducing power or has a positive oxidizing power.

2d. Less than 3 grams of lead reduced. If 1 A.T., assumed to be 30 grams for calculation, is taken for assay, the lead reduced will be the weight of the lead button from the preliminary assay multiplied by 6, so that if this is less than 3 grams, to give an 18-gram button we must add argol or charcoal to make up the deficiency.

3d. Three grams of lead reduced. This will give a button of the proper size if 1 A.T. of ore is used, i.e.,  $3 \times 6 = 18$ .

4th. More than 3 grams. Suppose the button weighs 6 grams, 1 A.T. of ore will give 36 grams, or 18 grams in excess, so enough nitre is added to oxidize 18 grams of lead, or  $\frac{1}{4} = 4.5$  grams. The oxidizing power of the nitre used must be determined, as already described.

Should the ore have an oxidizing power, which will be readily seen from its reddish or brown color, the following charge will show the amount of argol to be added: ore, 5 grams; PbO, 80 grams; soda, 20 grams; borax glass, 5 grams; argol, 3 grams; salt cover.

From the deficiency in the weight of the lead button as compared with the amount reduced by three grams of argol the oxidizing power of the ore is easily calculated and from this the quantity of reducing agent to add is estimated.

The reducing power of a sulphuret depends considerably on the character of the flux; if acid, the sulphur oxidizes to  $\text{SO}_2$ ; if basic, to  $\text{SO}_3$  and forms alkaline sulphates. In the preliminary assay the object is not to determine the theoretical reducing power, but the reduction under certain conditions, which should approach as nearly as possible those of the regular assay.

The following results were obtained according to the charge as given: \*

| Ore.                | Grams Pb Reduced<br>by 1 Gram. |
|---------------------|--------------------------------|
| Pyrite.....         | 11.60                          |
| Copper pyrites..... | 8.90                           |
| Zinc blende.....    | 8.30                           |
| Stibnite .....      | 6.20                           |
| Galena.....         | 3.35                           |

If the soda and borax glass are omitted, the lead reduced will be less, as already explained, and agree better with the calculated theoretical reduction.

The following table shows results obtained by use of preceding charge for preliminary assay:

---

\* Beringer's Assaying, page 98.

| Ore.                | Button<br>from<br>5 grams. | Calculated nitre,*<br>KNO <sub>3</sub> , for 18-<br>gram button. | Button obtained from<br>1 A.T. Ore, and nitre as<br>calculated. |
|---------------------|----------------------------|--|---|
| Sulphuret.....      | 22.90                      | 33.0   | 23.1  |
| Zinc blende.....    | 30.10                      | 41.0   | 21.3  |
| Zinc and copper.... | 14.76                      | 19.0   | 17.4 and 21.1   |
| Galena and pyrites. | 28.90                      | 41.80  | 16.5 and 17.0   |
| Antimonial.....     | 13.81                      | 17.36  | 20.88   |

*Roasting.*—Ores containing a large amount of sulphur, arsenic, antimony, or zinc should be roasted. In the former case if the ore is not roasted there will be danger of the formation of oxysulphurets, which, though fusible, are not decomposed at a white heat and enter the slag carrying silver with them. A large quantity of nitre is liable to boil over; even should this not occur, the evolution of vapors puffs up the mass and lead may be left adhering to the sides of the crucible. Arsenic and antimony produce arseniates and antimonates, which carry silver into the slag. Zinc also increases the loss of silver by volatilization and in the slag.

The ore may be roasted conveniently in a cast-iron pan over the furnace. The pan should be coated with red ochre or chalk, which protects it and helps to prevent loss of ore.

The weighed sample must be spread over the pan, and stirred until all danger of fusion is past. The ore must be heated gradually, not above a dull red for some time, and finally to a full red or higher heat. Too high a temperature at the outset causes the fusion of sulphides and the formation of matte troublesome to roast. A rapid dis-

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\* Oxidizing power of nitre used, 3.6 grams of lead. Some ores having a very high reducing power do not give such satisfactory results, the button obtained being less than would be expected.

engagement of arsenic, antimony, or zinc will also cause a mechanical loss of silver. Should fusion occur, it is better to weigh out a fresh portion of ore and roast it again. The operation may be considered finished when, after keeping the pan at a full red heat for some time, no fumes can be seen or smelled.

If copper pyrites be present, after roasting, cool, and mix some ammonium carbonate with the ore. Cover and heat the pan until fumes have ceased. The sulphates are converted into volatile ammonium sulphate, which passes off.

Arsenic and antimony require the addition of fine charcoal to reduce oxides or arseniates and antimoniates formed in roasting, care being taken to burn out all the charcoal. If the ore contains a fusible sulphide, such as antimony glance or galena, mix with some fine sand before roasting. Ores may be roasted in the muffle, in the earthen saucer already mentioned, page 23.

An ore which has been "dead" roasted should have no reducing power, and may have an oxidizing power due to the sesquioxide of iron formed in roasting, if the ore contained pyrites.

*Fusion.*—The prepared charge is thoroughly mixed and placed in a crucible. A hot fire is employed, and the crucible removed when complete fusion has taken place. This requires from thirty to forty-five minutes. The crucible is tapped gently on the floor, poured, or broken when cold.

After preparing a charge from the data obtained from the preliminary assay it should be fused, and the slag carefully examined before running a duplicate, so that silica

or borax glass may be added if the slag is basic, or any mistakes as to reducing power corrected.

(b) *Scorification Assay*.—This method is applicable to any rich ore, however impure, and is particularly valuable for sulphides rich in silver.

The reagents for scorification assay are pure granulated lead and borax glass.

An alloy of lead with the precious metals and a slag composed of litharge with the impurities and gangue of the ore are formed. The proportions of lead and borax will vary, and should be greater as the gangue and metallic oxides are more difficult of fusion. The following table shows the proportions found by experience to be adapted to the different ores; they are referred to one part of ore:

| Character of Ore.                 | Parts<br>Test-lead. | Parts<br>Borax Glass. |
|-----------------------------------|---------------------|-----------------------|
| Quartzose gangue.....             | 8-10                | 0.00                  |
| Basic gangue.....                 | 8-10                | 0.25-1.00             |
| Galena .....                      | 6                   | 0.15                  |
| Arsenical.....                    | 16                  | 0.10-0.50             |
| Antimonial.....                   | 16                  | 0.10-1.00             |
| Fahlerz (tetrahedrite).....       | 12-16               | 0.10-0.15             |
| Iron pyrites .....                | 10-15               | 0.10-0.20             |
| Blende.....                       | 10-15               | 0.10-0.20             |
| Copper.....                       | 10-20               | 0.10-0.15             |
| Nickel or cobalt.....             | 20                  | 0.15-0.20             |
| Tin.....                          | 20-30               | 0.15-0.25             |
| Telluride.....                    | 16-18               | 0.10                  |
| { and a cover of litharge (PbO) } |                     |                       |

In most cases one tenth of an assay ton of ore and thirty to forty grams of lead will be found to work well.

The ore and one half the lead are mixed in the bottom of the scorifier, and the rest of the lead poured over the mixture so as to form a cover, two or three lumps of borax glass the size of a pea being placed on top. The charge of ore varies from one third to one twentieth of an assay ton according to its richness, and if one scorifier will not contain it, weigh equal fractional parts for the number required, rather than to weigh the whole charge and roughly divide it between the scorifiers.

Four distinct periods may be noted in the working of an assay: (1) Melting. (2) Roasting. (3) Fusion. (4) Scorification.

(1) A strong heat is maintained at first to melt the lead. This is effected by closing the muffle and increasing the draft. As soon as the lead is fused the muffle is opened, and the ore is seen floating upon the surface of the lead. In a large muffle it is sufficient to place the scorifier in the back part first, and move it forward when the lead is fused.

(2) The roasting commences and is continued at a moderate heat until no more fumes are seen and the ore has disappeared.

(3) The heat is raised in order to fuse all the material. When the fusion is complete, clear white fumes of lead arise from the scorifier, there is a play of colors across the surface of the lead and the slag encircles the metallic bath like a ring. The borax glass plays an important part just here by giving liquidity to the slag, so that it can be thrown to the side as fast as formed, exposing the lead for oxidation. If borax glass is not added, and the ore contains much gangue and is not easily fusible, the scoriæ will float in masses over the lead, impeding the oxidation.

(4) When fusion is complete, the heat is lowered to a



constant point, until the ring of slag, which is continually growing smaller, closes over the lead. Then the heat should again be raised to liquefy the slag and allow the lead to settle, after which the scorifier is removed from the furnace, cooled or poured. Hammer the button as usual. The whole assay occupies from thirty-five to fifty minutes. Too much borax should not be added at first; it is better to add only a portion with the ore, and to introduce the rest as needed during the operation, wrapped in a small piece of paper.

The lead button from the scorification assay should be soft, malleable and weigh about twelve grams.

The scorification assay requires more attention and greater skill than the crucible assay. Sometimes masses of ore adhere to the sides of the scorifier and so escape the action of the litharge; these must be dislodged by shaking the scorifier with a rotary movement. If crusts of antimony oxide, etc., form, they may be removed by adding a pinch of charcoal.

When the scorification is proceeding satisfactorily, the slag is well fused, dark, and clear; the lead in the centre is bright. The heat should be as low as is consistent with perfect scorification. Pure ores will stand more heat than impure ones, such as zinc blende, stibnite, etc., without causing loss of silver by volatilization and in the slag.

If a scorification works badly, it usually needs more heat, lead or borax glass.

For very impure ores roasting preliminarily in the scorifier at a low heat before adding test-lead and borax glass will facilitate the subsequent operations.

*Scorification of the Lead Button.*—This process is not essential to the assay of many ores, and should be

avoided, for, besides requiring time, it introduces an extra operation and so causes another trifling loss of precious metals. It is necessary when the button is too large, hard or brittle, or where buttons from several assays are combined before cupellation. The object is either to reduce the size of the button or to remove some impurities which would interfere with the cupellation, or both.

To reduce the size of the button, place it in the scorifier with a pinch of borax glass and allow to scorify until the button is the proper size. It is not usually necessary to allow the slag to cover over.

If the button is the right size, but hard or brittle, about 20 grams of test-lead and a pinch of borax glass must be added.

If the button is large, say 40 to 50 grams, and impure, scorify with the addition of borax glass only.

Brittle buttons are caused by the presence of arsenic, antimony, zinc, litharge, sulphur, bismuth, mercury, selenium, tellurium, gold (in large amount) or platinum.

Hard buttons are caused by the presence of copper, iron, nickel, cobalt, platinum or a large amount of silver.

If matte is found over the button in the crucible assay, place this in the scorifier and roast thoroughly first, then add the button, test-lead and borax glass, and scorify.

Avoid reducing the size of a button from a rich ore to less than ten grams, for when the ratio of lead to silver becomes small the loss in scorification may exceed that of cupellation.

*Cupellation.*—This operation differs from scorification in that the scorix formed are absorbed by the cupel, leaving a pure bead of the precious metals.

A good cupel will absorb its own weight of litharge,

but it is better to use a cupel one third heavier than the button. The operation of cupelling is conducted as follows: A cupel is wiped out with the fingers carefully and all extraneous matter blown out; it is then placed in the muffle and heated until of the same temperature as the latter and all moisture and organic matter have been expelled, then the button, which should be malleable and the proper size, is gently placed in the cupel with a pair of forceps. The muffle is then closed by a door or a piece of lighted charcoal, to melt the lead. This done, the muffle is opened and the button, which at first appears bright and uncovered, is soon coated with a film of oxide moving in luminous patches over its surfaces and being continually thrown toward the edge, where it is absorbed by the cupel. The button gradually diminishes in size by oxidation and absorption and becomes more convex; the patches become larger and move more quickly; the last of the lead is absorbed, and the residue appears to revolve rapidly, becomes very brilliant, and is suffused with the tints of the rainbow and then presents the appearance of the precious metals. The latter part of the operation is called the "brightening" of the button. Should the bead be large and composed of silver, it must be removed slowly from the furnace to prevent "sprouting," by which portions of the metal are thrown off and lost. In case the bead is very large, say one hundred to three hundred milligrams, it is well to cover it with a hot cupel. If the bead is not larger than the head of an ordinary pin, the danger of sprouting is slight, and no great precaution need be taken in its removal.

Two causes have been assigned for sprouting. First, that molten silver absorbs oxygen from the air and gives it



up suddenly at the moment of solidification. Second, that by rapid cooling a crust is formed upon the outside of the button, which contracts upon the liquid interior and forces it out. Gold diminishes the tendency of buttons to sprout. When the gold is 40 per cent. of the silver there is no danger of loss in this way.

It is well to raise the heat of the muffle just at the time of brightening, or to push the cupel into the hotter part, to remove the traces of lead.

Silver is sensibly volatile at a high heat, and the loss increases with the temperature. We must avoid the two extremes of high heat and quick work, and low heat and prolonged work. The following are indices of favorable working: the muffle is reddish white, the cupel red, the fused metal luminous and clear, the lead fumes rise slowly and the litharge is completely absorbed by the cupel.

The heat is too great when the cupels are whitish, when the fused metal is seen with difficulty and the scarcely visible fumes rise rapidly.

The heat is too low when the fumes are thick and fall, and when the unabsorbed litharge forms lumps and scales about the button.

The degree of heat may be greater according as the lead is poorer in silver. By bearing this in mind the assayer can often hasten the operation without detriment.

Too strong a current of air cools the cupel and oxidizes the lead faster than it can be absorbed. Too slow a current prolongs the operation and increases the loss by volatilization.

Sometimes the material in a cupel becomes solidified in the midst of an operation, stopping further action. This

is called "freezing," and is occasioned by the production of litharge more rapidly than it can be absorbed by the cupel, infusible scoriæ due to a cold furnace or an excess of foreign oxides. It can sometimes be remedied by raising the heat of the muffle; or if the accident be due to foreign oxides, an addition of pure lead may be made to the assay. In either case the results are unreliable.

An assay that has passed well furnishes a bead well rounded, crystalline below and readily detached from the cupel. If the bead contains lead, it is brilliant below and does not adhere at all to the cupel. If it exhibits rootlets, the results are inaccurate and must be rejected.

Crystals of litharge—"feathers"—just inside the outer edge of the cupel indicate that the heat has been right.

The following table shows the difficulties which may arise in cupellation and how to guard against them :

| Button may        | Cause.  | Prevention.                                  | Remedy.   |
|-------------------|---|--|---|
| Spit.             | Volatile constituents, S, As, Sb, Bi, Hg, C.  | Hot cupel, malleable button.                 | Heat very slowly.                               |
| Freeze.           | Infusible substances, Fe, Ni, Co, Cu, Pt, Sn. | Good heat, malleable button, excess of lead. | Add Pb and raise temperature.                   |
| Sprout.           | Oxygen and contraction.                       | Careful removal and slow cooling.            | Add Pb and re-cupel.                            |
| Form rootlets.    | Imperfect cupel.                              | Good cupel.                                  | Dig out roots, wrap in lead and cupel again.    |
| Scorify.          | Hard cupel and too rapid cupellation.         | Good cupel.                                  | Raise temperature and make atmosphere reducing. |
| Sink.             | Cracked cupel.                                | Stop cupellation.                            | Remove and use fresh cupel.                     |
| Become encrusted. | Rapid oxidation.                              | Slow work or scorify button.                 | Add Pb and regulate temperature.                |

*Weighing the Gold and Silver Bead.*—The bead of gold and silver is detached from the cupel with pincers,

thoroughly cleansed with a small stiff brush, and weighed on the bullion-balance.

*Inquartation and Parting.*—The separation of gold from silver is termed parting. It is effected by means of nitric acid, which dissolves the silver and leaves the gold. It is essential that a certain relation should exist between the amount of gold and silver in the alloy. If there be too little silver, it will not dissolve completely, but will be so enveloped in the gold as to escape the action of the acid. If too much silver be present, the gold obtained will be so fine and light as to occasion loss in washing.

The amount of silver present should be from two to three times the weight of the gold. The assayer must judge by the color of the bead as to the proportion of silver contained, and if it be too small he must supply the deficiency with pure silver, which is kept on hand in thin foil. The bead and silver are well fused together to effect complete distribution of the silver. The fusion may be made on charcoal by the blowpipe or by wrapping the bead and silver in a cornet of lead-foil and cupelling.


The bead is then flattened on an anvil, and treated in a porcelain capsule (Fig. 36) with nitric acid, C.P. 1.16 sp. gr. (21° Baumé). Enough acid is added to cover the bead and heated gently. The acid must  be free from chlorine, which would precipitate the silver. When all action of the first acid has ceased, decant and carefully add some fresh acid of 1.26 sp. gr. (32° Baumé). Heat for several minutes, pour off the acid, wash thoroughly with distilled water and dry the residue of gold. It is well to apply a high heat before attempting to remove the gold, to render it adherent. The

FIG. 36.

gold residue is detached with a knife, transferred to a cornet of lead, cupelled and weighed; or if perfectly clean and yellow, weighed without cupellation.

In parting neither allow the acid to act for a long time in the cold, nor boil violently, as the gold will be separated in a finely divided state, which is more difficult to wash without loss.

When drying the gold, before annealing, turn the capsule so that the last drop of water is on the opposite side from the gold and thus avoid spattering and consequent loss.

*Weighing the Gold.*—The gold obtained is weighed as described and the assay is completed.

*Calculation of Results and Reporting.*—The milligrams of precious metals obtained per assay ton of ore, after deducting the silver contained in the test-lead or litharge, correspond to the Troy ounces in a ton of two thousand pounds, avoirdupois. There is therefore no trouble save in the case of an ore which contains metallic scales and the method to be employed when such is the case can be shown by an example.

The sample presented for assay weighed 485 gms. Pulverized in a mortar to flatten the scales and sifted in a box sieve (Fig. 26), it gave:

- (a) Sifted ore..... 476 gms.
- (b) Metallic scales..... 5 “

In calculating the results of the assay the weight of the sample minus the scales is considered siftings, as no scales should be lost in pounding or sifting.

(a) *Sifted Ore.*—Ten grams gave by crucible assay:

Gold..... 4.0 mgs.  
Silver, after deduction of the silver in the litharge. 14.3 “

Hence the total precious metals in the siftings is :

$$\text{Gold} \dots\dots\dots \frac{4.00}{10} \times 480 = 192.0$$

$$\text{Silver} \dots\dots\dots \frac{14.30}{10} \times 480 = 686.4$$

(b) *Metallic Scales*.—These melted with lead gave a button of 60 grams, which was rolled out and 10 grams taken for cupellation. This yielded :

$$\text{Gold} \dots\dots\dots 2.6 \text{ mgs.}$$

$$\text{Silver} \dots\dots\dots 500.0 \text{ “}$$

Hence the total precious metals in the scales must be :

$$\text{Gold} \dots\dots\dots \frac{2.6}{10} \times 60 = 15.60 \text{ mgs}$$

$$\text{Silver} \dots\dots\dots \frac{500.0}{10} \times 60 = 3000.00 \text{ “}$$

Total :

$$\text{Gold in siftings} \dots\dots\dots 192.00 \text{ mgs.}$$

$$\text{“ “ scales} \dots\dots\dots 15.60 \text{ “}$$

$$\text{“ “ ore taken} \dots\dots\dots 207.60 \text{ “}$$

Hence  $\frac{207.60}{485} \times 29.166$  (value of an assay ton) = gold per assay ton of original ore.

$$\text{Silver in total siftings} \dots\dots\dots 686.40 \text{ mgs.}$$

$$\text{“ “ scales} \dots\dots\dots 3000.00 \text{ “}$$

$$\text{“ “ ore taken} \dots\dots\dots 3686.40 \text{ “}$$

Hence  $\frac{3686.40}{485} \times 29.166$  (value of an assay ton) = silver per assay ton of original ore.



## CHARGES FOR SPECIAL ORES.

*Pure Ore, Quartz Gangue.*—(1) Rich: (a) Crucible assay. Ore, 1 A.T.; PbO, 2 A.T.; soda, 1 A.T.; argol,  $1\frac{1}{2}$  grams; borax glass, 10 grams; salt cover.

(b) Scorification assay. Ore,  $\frac{1}{2}$  A.T.; test-lead, 40 grams; borax glass,  $\frac{1}{2}$  gram.

(2) Low grade: Ore, 2 A.T.; PbO, 2 A.T.; soda, 2 A.T.; argol,  $1\frac{1}{2}$  grams; borax glass, 10 grams; salt cover.

(3) Tailings: Ore, 4 A.T.; PbO, 2 A.T.; soda, 4 A.T.; argol, 2 grams; borax glass, 10 grams; salt cover.

*Pure Ore, Basic Gangue.*—Same charges, with the addition of from 1 to 3 A.T. of SiO<sub>2</sub>. If the gangue is magnesite or barite, increase both the soda and silica.

*Galena.* (a) Scorification assay: Ore,  $\frac{2}{3}$  A.T.; test-lead, 40 grams; borax glass,  $\frac{1}{2}$  gram.

(b) Crucible assay: Ore, 1 A.T.; PbO, 1 A.T.; soda, 1 A.T.; nitre, 20 grams; borax glass, 10 grams; salt cover.

*Zinc Blende or Sphalerite.*—(a) 10 to 25 per cent. zinc: Ore,  $\frac{1}{2}$  A.T.; test-lead, 60 grams; borax glass, 1 gram.

(b) 25 to 50 per cent. zinc, 2 portions each: Ore,  $\frac{1}{10}$  A.T.; test-lead, 60 grams; borax glass, 1 gram. Combine the buttons and rescorify. A low heat must be used in the scorification of this ore.

*Arsenopyrite, Mispickel.*—(a) Crucible assay: Roast 2 A.T. of ore; then add PbO, 3 A.T.; soda,  $1\frac{1}{2}$  A.T.; silica, 2 A.T.; borax glass, 10 grams; argol,  $2\frac{1}{2}$  grams; salt cover.

(b) Scorification assay: Run 3 portions. Ore,  $\frac{1}{2}$  A.T.; test-lead, 60 grams; borax glass, 2 grams. Combine the buttons and rescorify.

*Stibnite.*—For gold, crucible assay: Mix 1 A.T. ore with

1 A.T. clean sand and roast; then add a large spoonful of ground charcoal and heat until no more sparks are seen; mix with PbO, 2 A.T.; soda, 1 A.T.; argol, 2 grams; borax glass, 10 grams; salt cover.

For silver, scorification assay: Ore,  $\frac{1}{2}$  A.T.; test-lead, 70 to 80 grams; borax glass, 1 gram. Scorify at a low heat; add charcoal if needed.

*Gray Copper, Tetrahedrite, Fahlerz.*—Scorification assay: Ore,  $\frac{1}{2}$  A.T.; test-lead, 80 grams; borax glass,  $\frac{1}{2}$  gram.

Crucible assay as given for arsenopyrite. In either case the buttons may need rescorifying to remove impurities before cupellation.

*Pyrite, Iron Pyrites.*—Crucible assay: (1) Roast 2 A.T.; then add PbO, 3 A.T.; soda, 2 A.T.; silica, 2 A.T.; argol:  $3\frac{1}{2}$  grams; borax glass, 10 gram; salt cover.

(2) Ore, 1 A.T.; litharge, 2 A.T.; soda, 2 A.T.; silica,  $1\frac{1}{2}$  A.T.; nitre, 50 grams; salt cover.

(3) Ore, 1 A.T.; PbO, 1 A.T.; potassium cyanide,  $2\frac{1}{2}$  A.T.; salt cover. Moderate fire.

For ores containing but little pyrites use one of the charges given for a pure ore, with the addition of a few nails or wires. This method of desulphurizing is not recommended for ores containing much sulphur or concentrates.

*Chalcopyrite, Copper Pyrites.*—Roast with addition of ammonium carbonate and use charges given for iron pyrites. The buttons may require scorifying to remove copper.

*Oxide of Iron, Hematite, etc.*—See charge (1) under Pyrite.

*Tellurides.*—Rich: (1) Scorification assay: Ore,  $\frac{1}{2}$  A.T.;

test-lead, 70 grams; borax glass,  $\frac{1}{2}$  gram; cover of litharge (20 grams).

(2) Crucible assay: Ore,  $\frac{1}{2}$  A.T.; PbO, 4 A.T.; soda, 1 A.T.; silica, 1 A.T.; argol,  $1\frac{1}{2}$  grams; borax glass, 10 grams; salt cover.

Low grade: Ore, 2 A.T.; PbO, 4 A.T.; soda, 2 A.T.; silica,  $1\frac{1}{2}$  A.T.; argol, 2 grams; borax glass, 10 grams; salt cover.

Scorify the buttons if brittle. Cupel at a low heat near the end, as the button has a tendency to separate into small particles.

The preceding charges have worked satisfactorily in the assay laboratory of Columbia University, but it is not to be inferred that other charges will not give equally good results.

REMARKS.—The assayer must always modify the charge to suit the particular ore he is testing. An examination of the slag and button will often suggest what change to make.

Too small a button, less than 10 grams, will not ensure the collection of all the gold and silver; hence the reducing agent must be increased.

A brittle button shows the necessity of more desulphurizing by roasting, nitre, etc.

An earthy slag shows an insufficient quantity of silica.

No attempt has been made to give charges for ores containing mixtures of different minerals, as these could not possibly be made so as to cover every case. The assayer must, by a combination of the nearest charge, intelligence, and experiment deduce the proper proportions of ore, fluxes, reducing agents, etc.

## ACCURACY OF GOLD AND SILVER ASSAYS.

The fire assay for gold and silver is the most accurate and satisfactory method for their determination in most cases; there are, however, small losses of the precious metals in each operation.

The inaccuracies, aside from sampling, are as follows:

1st. Loss in the slag, crucible or scorification.

2d. Loss by absorption in the cupel.

3d. Loss by volatilization.

4th. Excess in weight of bead due to imperfect removal of base metal in cupellation.

5th. Excess in weight of the residue due to imperfect solution of the silver by nitric acid, some silver being retained by the gold, and consequently after annealing being weighed as gold.

The 4th and 5th inaccuracies can be avoided by careful work, as already described. The loss by volatilization cannot be eliminated entirely, but should be reduced to a minimum by cupelling at a low heat (with feathers), and by roasting all ores containing zinc before making a crucible fusion.

The 1st and 2d losses can be almost entirely recovered by making a corrected assay—that is, by grinding and reassaying the slag and cupel, and adding the gold and silver recovered to that obtained in the regular assay.

The correction may be carried further by making a second correction on the cupel and slag from the first, but the amount recovered will not appreciably affect the result.

The loss by volatilization during cupellation has been

worked out carefully for silver bullion (see page 121), but it is preferable to determine this correction for each particular case, by running a charge containing a known amount of silver under the same conditions as the assay to be corrected, and then adding the loss in the test assay.

This plan must be used with rich ores where a varying amount of impurity in the lead button causes a considerable difference in the loss by volatilization.

The losses in assaying ores, as determined, are as follows :

**GOLD.**—The loss of gold in the assay of any ore except when extremely rich is too small to require correction.

In a rich argentite containing 241 ounces of gold this loss was as follows : \*

|                                    |          |
|------------------------------------|----------|
| Recovered from crucible slag ..... | 0.0515%  |
| “ “ cupel.....                     | 0.0725 “ |
| Correction for crucible assay..... | 0.1240 “ |
| “ “ scorification assay.....       | 0.3100 “ |

Furman † gives the average loss of gold in the crucible assay, determined by difference on the pure metal, as 0.3%.

**SILVER.**—The loss of silver is much greater than of gold. The absolute loss increases with the richness of the ore, but the percentage of loss diminishes.

The following results were obtained from a cerussite carrying 2260 ounces of silver :

| Method.               | Recovered<br>from Slag. | Recovered<br>from Cupel. | 1st<br>Correction. | 2d<br>Correction. | Total.  |
|-----------------------|-------------------------|--------------------------|--------------------|-------------------|---------|
| Scorification . . . . | 2.804%                  | 1.398%                   | 4.202%             | 0.540%            | 4.742%  |
| Crucible.....         | 0.927 “                 | 1.109 “                  | 2.036 “            | 0.252 “           | 2.288 “ |

\* Miller and Fulton, S. of M. Quarterly, Vol. XVII., p. 169.

† Trans. A. I. M. E., Vol. XXIV., p. 735.

F. P. Dewey \* gives the following table showing the inaccuracy in assaying different material :

|                     | Value.      | Range of Inaccuracy. | Average Inaccuracy. |
|---------------------|-------------|----------------------|---------------------|
| Tailings.....       | 1 to 20 oz. | 18 to 8.6 %          | 13.7%               |
| Washed ore .....    | 15 " 35 "   | 13 " 10 "            | 10.4 "              |
| Raw ore.....        | 20 " 50 "   | 16 " 5 "             | 9.7 "               |
| Roasted ore.....    | 20 " 45 "   | 12 " 6 "             | 9.1 "               |
| Lead carbonate..... | 50 " 100 "  | 5.1 " 2.6 "          | 3.8 "               |

Comparison of scorification and crucible assays for silver in lead ores containing zinc : †

| Method.             | Commercial Assay. | Corrected Assay. | Ore Contained. |
|---------------------|-------------------|------------------|----------------|
| Scorification ..... | 13.80             | 13.80            | { 18.5% Pb     |
| Crucible .....      | 12.20             | 13.98            |                |
| Scorification.....  | 29.40             | 29.97            | { 46.3" Pb     |
| Crucible .....      | 28.92             | 30.46            |                |
| Scorification ..... | 13.00             | 13.50            | { 14.6" Pb     |
| Crucible. ....      | 12.80             | 13.60            |                |

The fact that scorification gives the highest uncorrected assay agrees with our own experiments. Uncorrected scorification assays are higher than uncorrected crucible assays of impure materials (sulphides, etc.) carrying silver.‡ The corrected crucible assay is usually slightly higher than the corrected scorification assay, because of the loss of silver in scorification by volatilization.

CHARGES FOR CORRECTED ASSAYS.—Cupels give a very

\* J. Am. Chem. Soc., Vol. XVI., p. 513.

† C. A. Stetefeldt, Trans. A. I. M. E., Vol. XXIV., p. 530.

‡ S. of M. Quarterly, Vol. XVII., p. 169.

pasty fusion on account of the phosphates present. The following charge has been used with success. For one cupel (crucible assay) PbO, 2 A.T.; soda, 1 A.T.; borax glass, 1 A.T.; argol, 2 grams.

*Crucible Slags.*—Add PbO, 1 A.T.; argol, 2 grams; and remelt.

*Scorification Slags* need only some borax glass and argol.

In making a corrected assay it is not often necessary to determine the amount recovered from the cupel and slag separately. They are ground together and melted in a crucible with a charge containing borax glass, like the following: PbO, 2 A.T.; soda, 2 A.T.; borax glass, 1.5 A.T.; argol, 2.5 grams.

## GOLD AND SILVER IN COPPER MATTES.

A copper matte is an artificial sulphide of copper and iron usually containing gold and silver, and often lead, antimony, zinc, bismuth, etc.

The determination of gold and silver is of special importance, as great quantities of matte are sold on the results of assays. Corrected assays are often made; and in the absence of an agreement between the buyer and seller as to the method of assay, the assayer should state the method used, and whether “regular” or “corrected.”

**DRY METHODS.**—(1) Weigh out four portions of  $\frac{1}{16}$  A.T. each on an accurate balance, mix each with 40 grams of test-lead, transfer to scorifiers, cover with 40 grams more test-lead, and place half a gram of borax glass on top. Scorify at a very low heat near the mouth of the muffle until covered over, then raise heat, and pour as usual into

moulds. Hammer the buttons. Combine two buttons and scorify with the addition of a little borax glass. The resulting lead button should be malleable and weigh about 12 grams. If it shows the presence of copper, it must be rescorified with test-lead until malleable. Cupel with feathers, weigh, part, using first some dilute nitric acid sp. gr. about 1.14, then 1.26 sp. gr. Weigh the gold. Test the nitric acid solution of silver for copper, which must be deducted if found to be present. This method gives duplicate results on  $\frac{1}{4}$  A.T., and is accurate for the gold, but low for silver. To correct, combine the cupels, grind, and make a crucible assay as usual for silver.

(2)\* Weigh out ten portions of  $\frac{1}{10}$  A.T. each, scorify in a  $2\frac{1}{2}$ -inch scorifier, mixing with 30 grams test-lead and  $\frac{1}{4}$  gram borax glass, and covering with 30 grams of lead and 1 gram borax glass. Start with a bright red heat; continue at a little lower heat until the end, then raise to a bright red heat and pour. Clean off slag, transfer to new scorifiers and rescorify. Cupel each lead button separately, and weigh the ten resulting silver-gold beads together. Grind up and fuse cupels in five lots of two each, and cupel the buttons, adding the five resulting beads to the ten silver-gold beads. Charge for fusing two cupels by crucible assay: soda,  $\frac{1}{4}$  A.T.; PbO,  $2\frac{1}{2}$  A.T.; borax glass, 1 A.T.; argol, 2.5 grams. Mix thoroughly with ground cupels, and add either salt or borax glass as a cover. Part the buttons with nitric acid 1.14 followed by 1.42 sp. gr. Boil until the acid becomes colorless, wash, and weigh as usual.

This is a slight modification of the method used at the Omaha & Grant Smelter.

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\* Trans. A. I. M. E., 1895: Assays of Copper and Copper Mattes.



**COMBINATION METHOD.\***—One A.T. of copper matte is placed in a No. 5 beaker or a large casserole and treated with 100 c.c. of water and 50 c.c. of nitric acid 1.42 sp. gr. and covered; when the first action has ceased, 50 c.c. more acid is added, and the solution heated until the decomposition is complete. Most of the free acid is boiled out; then 400 c.c. water, 3 c.c. sulphuric acid, and 10 c.c. of a saturated solution of lead acetate are added. The dense white sulphate of lead carries down with it the finely divided gold. Allow the precipitate to settle, filter, and wash once with water, dry, transfer to a scorifier, and burn up the paper and any unoxidized sulphur, in the front of the muffle, then add 40 grams of test-lead and scorify as usual. (If there is much gold in the matte, before filtering add 1 c.c. normal salt solution (equals 10 mgs. Ag) to precipitate enough silver to avoid inquartation.) Cupel as usual.

To the filtrate add more acetate of lead, sulphuric acid, and a slight excess of salt solution, filter, dry, ignite, add lead, scorify, and cupel as usual. Weigh the buttons separately, part the first and add the silver obtained to the weight of the second. All the gold will be in the first button.

**REMARKS.**—The advantages of the treatment with nitric acid are the removal of copper, zinc, etc., which give trouble in scorification and cupellation, the diminished loss of silver by absorption in the cupel, and also the use of a larger portion of the matte.

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\* Trans. A. I. M. E., Vol. XXIV., page 576.

The loss of silver in the slag and by volatilization is not usually determined.

For a more complete discussion of the assay of copper mattes see Trans. American Institute of Mining Engineers for 1894 and 1895.

The combination and dry methods agree well for silver, if correction for loss in the cupel is made; the gold is usually lower by the combination method, as is shown by the following results :\*

ASSAYS OF COPPER MATTE.

| Scorification method :             | Silver,<br>Ounces per Ton. | Gold,<br>Ounces per Ton. |
|------------------------------------|----------------------------|--------------------------|
| Average of results by 14 assayers. | 128.86                     | 2.286                    |
| Combination method :               |                            |                          |
| Average of results by 10 assayers. | 127.25                     | 2.212                    |

ASSAY OF PIG COPPER FOR GOLD AND SILVER.†

GOLD.—The gold is most accurately determined by scorifying 6 to 10 portions of  $\frac{1}{10}$  A.T. each with 50 grams of test-lead and combining the silver-gold buttons after cupellation. The following method, or some modification of it, is usually employed : Weigh out two portions of 1 A.T. each, into large beakers, add 100 c.c. distilled water, then 50 c.c. nitric acid, sp. gr. 1.42, to each and cover. After

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\* Trans. A. I. M. E., March, 1895.

† Method D, Trans. A. I. M. E., March, 1895.

the violent action is over add 50 c.c. more acid and warm till nothing more dissolves. Boil out part of the nitric acid, dilute each to 500 c.c. with distilled water, add 3 c.c. concentrated sulphuric acid and 10 c.c. of a strong solution of lead acetate. Stir vigorously and allow to settle for several hours, warm, filter, wash out the beakers with hot water and then wipe with filter-paper; add this to the paper containing the precipitate. Dry the filters and wrap them in about 8 grams of lead-foil, place in scorifiers and add to each 40 grams of test-lead and  $\frac{1}{2}$  gram of borax glass; start the scorification at a low heat, then scorify and cupel as usual. The beads will probably contain sufficient silver to part; if this is not the case, a small piece of pure silver should be placed in each cupel before the cupellation is begun. Part and weigh the gold as usual.

SILVER.—Treat in large beakers three portions of  $\frac{1}{3}$  A.T. each with half the quantity of water and nitric acid used in the gold assay. Proceed as already described, but before adding the lead acetate add just enough salt solution to precipitate the silver, avoiding very carefully a large excess. Cupel at as low a temperature as possible with “feathers.” Assay the cupels and scorification slags as already described if a “corrected” assay is desired.

REMARKS.—The following figures show results by different methods on a sample of copper borings:

| SCORIFICATION ASSAY.   |   |  |       | COMBINATION ASSAY.                                 |                            |                                |       |
|--|---|--|-------|--|----------------------------|--------------------------------|-------|
| SILVER AND GOLD.   |   |  | Gold. | SILVER AND GOLD.                                   |                            |                                | Gold. |
| First Obtained.  | Cupels and Slags Absorbed.                            | Total.   |       | First Obtained.                                    | Cupels and Slags Absorbed. | Total.                         |       |
| 156.90 }<br>164.00 }<br>156.80 }<br>150.70 }<br>156.90 }<br>154.80 }<br>155.60 }<br>150.80 }<br>157.80 }<br>151.90 } | 7.50<br><br>10.80<br><br>8.90<br><br>9.30<br><br>8.90 | 167.95 }<br>164.55 }<br>164.75 }<br>162.50 }<br>163.75 } | 0.35  | 156.20 }<br>156.20 }<br>155.72 }                   | 4.98                       | 161.02<br><br>0.24<br><br>0.24 |       |
| Average silver and gold... 164.70<br>Less gold..... 0.35   |   |  |       | Silver and gold ..... 161.02<br>Less gold.... 0.24 |                            |                                |       |
| Silver..... 164.35<br>Less copper..... 8.80  |   |  |       | Silver..... 160.78                                 |                            |                                |       |
| Silver, corrected..... 160.55  |   |  |       | No copper in silver beads.                         |                            |                                |       |

### ASSAY OF SILVER SULPHIDES.

The precipitated sulphides obtained by the Russell process frequently contain from 12,000 to 16,000 oz. of silver per ton, so that extraordinary care is required in assaying them.

(a) THE COMBINATION METHOD as given under copper matte may be used, with the following modifications: \* Take two portions of  $\frac{1}{10}$  or  $\frac{1}{15}$  A.T. each, instead of 1 A.T., and before scorifying mix with PbO 8 grams and test-lead 20 grams, instead of using all test-lead.

\* Furman, Trans. A. I. M. E., 1895: Assay of Silver Sulphides.

(b) DRY METHOD.—Run ten scorification assays at a low heat, using the following charge: sulphides,  $\frac{1}{16}$  A.T. (weighed most accurately); test-lead, 40 grams; borax glass,  $\frac{1}{2}$  gram. Assay the cupels and slags in sets of two, add the weight of silver recovered to the combined weight of the ten buttons, and multiply by two.

Great care must be taken to prevent the large silver buttons from sprouting.

The average correction for silver in precipitated sulphides, 2000 to 16,000 ounces, is given by Dewey as 1.7 %.

REMARKS.—The following average results were obtained in the assay laboratory of Columbia University on a sample of argentite:

Scorification assay:

|                                   |              |
|-----------------------------------|--------------|
| Corrected value, silver.....      | 17,363.9 oz. |
| Recovered from cupel, silver..... | 0.679 %      |
| “ “ slag “ .....                  | 0.754 “      |
| “ second correction, silver.....  | 0.222 “      |
| Total correction, silver.....     | 1.655 “      |

Crucible assay:

|                                    |              |
|------------------------------------|--------------|
| Corrected value, silver.....       | 17,268.4 oz. |
| Recovered from cupel, silver ..... | 0.730 %      |
| “ “ slag “ .....                   | 0.179 “      |
| “ second correction, silver.....   | 0.082 “      |
| Total correction, silver.....      | 0.991 “      |

The above methods of assay of rich sulphides also apply to the assay of “slimes” containing gold and silver, separated in the electrolytic refining of copper.

## ASSAY OF SWEEPS.

Sweeps are metallic clippings, filings, etc., mixed with dust and dirt, obtained by sweeping up the floors of workshops where the precious metal and their alloys are used. Besides organic matter they may contain gold, silver, platinum, brass, copper, lead, tin, iron, etc.

The principal difficulty in assaying sweeps, aside from the interference of platinum, is in securing an average sample.

ASSAY.—Weigh the entire sample, dry at about 120° C., and determine the loss. Take 2 to 6 A.T. and burn off the carbonaceous material in the muffle, in a roasting-dish or scorifier, avoiding a strong draft. When cold, transfer to an iron mortar and pound to flatten out the scales. Sieve through a 100-mesh sieve. If a large quantity of scales are left on the sieve, melt them in a scorifier with 60 to 100 grams of test-lead, and pour. Weigh this button and take weighed portions for assay, (see Lead Bullion). If but few scales are obtained, they may all be scorified with test-lead and cupelled.

The siftings are best run like an ore by crucible assay, in duplicate, using 15 to 25 grams of siftings, 2 A.T. litharge, 1 A.T. soda, 1 A.T. borax, and 2 grams of argol. Cupel the buttons as usual, and calculate the precious metals as given under Scale Ore.

For the interference of platinum see chapter on the Assay of Platinum.

REMARKS.—A sample of sweeps assayed as described gave:

|             |                         |
|-------------|-------------------------|
| Gold.....   | 11.94 oz. and 11.83 oz. |
| Silver..... | 67.35 oz. and 67.30 oz. |

For sweeps rich in silver a correction should be made for loss in slags and cupels.

### LEAD BULLION.

Lead or "base" bullion often contains, besides gold and silver, considerable quantities of copper, antimony, zinc, etc. The amount of these impurities determines the method of assay.

(a) PURE BULLION.—Weigh out two portions of  $\frac{1}{2}$  A.T. each, hammer into a cube, so that it will fit the cupel, and cupel direct at a low heat (with feathers); weigh, part with the two strengths of nitric acid, 1.16 and 1.26 sp. gr., anneal, and weigh the gold as usual. The duplicates should agree very closely, within 0.05 oz. for gold and 0.5 oz. for silver.

(b) IMPURE BULLION.—Scorify  $\frac{1}{2}$  to 1 A.T. with from 20 to 60 grams of test-lead and  $\frac{1}{2}$  to 1 gram of borax glass, depending on the amount of Cu, Sb, Zn, etc., present. With a rich bullion do not scorify the button down to less than 10 grams. If the button is hard, rescorify with more test-lead. Cupel, part, etc., as with pure bullion.

The loss in assaying base bullion varies from 1.5 to 5% under the most favorable conditions, and when cupelled at a high temperature or without previous scorification, if impure, may reach 8 to 12%.

Part of this loss can be recovered as usual.

### SILVER BULLION.

There are two methods in use for the determination of silver in coins and bullion: 1st. Cupellation method, used

largely in the West; preferable for very impure bullions, especially those containing mercury. 2d. Gay-Lussac or volumetric method, used by the mints and United States assay offices, and in some works. This method is the most accurate, and is fully described in Part III.

**CUPELLATION METHOD.** (*a*) *Preliminary Assay.*—The object is to determine the approximate fineness, so as to know how to make up a check assay having as nearly as possible the exact composition of the bullion. This check is cupelled under the same conditions as the regular assay, and from its loss of silver the correction for loss by volatilization and by absorption is obtained.

Weigh out 400 to 500 milligrams of the bullion, wrap it up in 5 to 10 grams of lead-foil, and cupel with the usual precautions, so as to give feathers of litharge on the edge of the cupel. Before removing from the muffle cover with a hot cupel, and then allow to cool gradually, as there is great danger of sprouting. If the button sprouts, the assay must be repeated. The button is cleaned, weighed, and parted for gold as usual.

Suppose 500 milligrams of the alloy gave a button of silver weighing 434.75 mgs., then  $500 : 1000 :: 434.75 : x = 869.5$ , approximate fineness. Corrections for loss of silver by cupellation can then be made from the following table. They are given in thousandths, and must be added to the standard.

| Standard. | Correction. | Standard. | Correction. | Standard. | Correction. |
|-----------|-------------|-----------|-------------|-----------|-------------|
| 998.97    | 1.03        | 670.27    | 4.73        | 346.73    | 3.27        |
| 973.24    | 1.76        | 645.29    | 4.71        | 322.06    | 2.94        |
| 947.50    | 2.50        | 620.30    | 4.70        | 297.40    | 2.60        |
| 921.75    | 3.25        | 595.32    | 4.68        | 272.42    | 2.58        |



| Standard. | Correction. | Standard. | Correction. | Standard. | Correction. |
|-----------|-------------|-----------|-------------|-----------|-------------|
| 896.00    | 4.00        | 570.32    | 4.68        | 247.44    | 2.56        |
| 870.93    | 4.07        | 545.32    | 4.68        | 222.45    | 2.55        |
| 845.85    | 4.13        | 520.32    | 4.68        | 197.47    | 2.55        |
| 820.78    | 4.22        | 495.32    | 4.68        | 173.88    | 2.12        |
| 795.70    | 4.30        | 470.50    | 4.50        | 148.30    | 1.70        |
| 770.59    | 4.41        | 445.69    | 4.31        | 123.71    | 1.29        |
| 745.38    | 4.52        | 420.87    | 4.13        | 99.12     | 0.88        |
| 720.36    | 4.64        | 396.05    | 3.95        | 74.34     | 0.66        |
| 695.25    | 4.75        | 371.39    | 3.61        | 49.56     | 0.44        |

**Example:** The number in the column of standards nearest to 869.5 is 870.93; the corresponding correction is 4.07; adding this to 869.5, we obtain 873.57 for the true approximate fineness.

We must also determine the base metal present: to do this dissolve some of the bullion in nitric acid, dilute and test for lead by sulphuric acid; then if it is present, so that the loss in cupellation is not due entirely to copper, determine the copper by one of the volumetric methods (given in Part III.) after precipitating the silver as chloride.

When the approximate composition is known, make up a test or check piece of 500 milligrams from pure silver,\* copper, etc., having this composition, and wrap in the amount of sheet lead required to give perfect cupellation. (See table, page 123.)

(b) *Regular Assay.*—Two portions of 500 milligrams each of bullion are weighed out on a bullion-balance as accurately as possible,† and wrapped up in the proper amount of sheet lead. The lead-foil should either be free

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\* For the preparation of pure silver see p. 43.

† Be careful to record the exact amount of bullion taken if it is not precisely 500 milligrams.

from silver or its exact contents known. Heat three cupels in the muffle until all carbonaceous material has been burned out; then introduce the assays, the check assay in the centre. The three assays must be run together, so that the heat, draft, etc., are the same. Weigh the three buttons. The difference between the weight of the button from the test assay and the weight of silver taken gives the loss of silver in cupellation. This loss is added to the average weight of the other two buttons; the sum multiplied by 2 equals the fineness (when exactly 500 mgs. are taken).

REMARKS.—The buttons from the bullion should not differ by more than 1 milligram. The loss in weight of the test assay should not be over 5 milligrams except with very impure bullion.

If gold is present, the buttons must be parted and the gold deducted. Platinum in small quantity will not be noticed, but will count as silver.

#### WEIGHTS OF SHEET LEAD FOR CUPELLATION.

| Fineness.        | Grams Lead for $\frac{1}{2}$ Gram of Bullion. |
|------------------|---|
| 950 to 1000..... | 5   |
| 900 " 950 .....  | 7   |
| 850 " 900 .....  | 8   |
| 800 " 850 .....  | 10  |
| 750 " 800 .....  | 11  |
| 700 " 750 .....  | 12  |
| 650 " 700 .....  | 13  |
| 600 " 650 .....  | 15  |
| 550 " 600 .....  | 16  |
| 500 " 550 .....  | 17  |

## GOLD BULLION.

| Fineness.       | Grams Lead for $\frac{1}{4}$ Gram of Bullion. |
|-----------------|---|
| 450 to 500..... | 18  |
| 400 " 450.....  | 19  |
| 300 " 400.....  | 20  |

## GOLD BULLION.

The assay of gold coin and bullion comprises two determinations: *a.* Base metal. *b.* Gold. The difference between these two and the total weight of bullion gives the amount of silver.

*a.* BASE METAL: *Cupellation*.—Weigh out 500 mgs. and cupel with ten times its weight of pure lead rolled out into a sheet, the bullion being wrapped in it. If the bullion contain much copper, use more lead, or one half the amount of bullion. (See table.)

The copper is oxidized and carried into the cupel by the litharge, leaving a button of gold, and silver if there be any present.

A check assay is made with every set of assays. Alloys for check assays are made up, having a composition similar to that of the bullion. The U. S. mint employs a proof-alloy containing 850 parts of gold, 12 parts of copper, and the rest silver. This ought to lose by cupellation just 12 parts of copper. It may lose more or less, and according to the difference one way or the other the regular assays which have been made under the same conditions are corrected. Suppose the check assay gave 11.8 copper instead of 12, the proportion of copper obtained in each of the regular assays must then be increased by 0.2 thousandths, or *vice versa*.

## PROPORTIONS OF LEAD FOR CUPELLATION.

| Bullion. |          | Parts Lead for 1 Part Bullion. |
|----------|----------|--------------------------------|
| Gold.    | Copper.  |                                |
| 950..... | 50.....  | 6                              |
| 900..... | 100..... | 12                             |
| 800..... | 200..... | 18                             |
| 700..... | 300..... | 24                             |
| 600..... | 400..... | 30                             |

This table gives the amounts of lead necessary to remove all copper except a trace. If there is much copper present, it is preferable to remove it by several cupellations with moderate quantities of lead.

*b. GOLD.\**—The determination of gold may be divided into five operations :

- (1) Preparation of the assay piece.
- (2) Cupellation.
- (3) Preparation for parting.
- (4) Parting and annealing the cornets.
- (5) Weighing and reporting.

(1) The sample is flattened with a hammer or rolled to about  $\frac{1}{32}$  of an inch in thickness ; then about  $\frac{1}{2}$  gram is cut off with shears (to within 1 milligram). Then weigh the sample most accurately, add  $1\frac{1}{2}$  grams of silver-foil free from gold, and wrap both in 5 grams of lead-foil.

If the gold is nearly 1000 fine, from 10 to 25 milligrams of copper are added. The amount of lead-foil to be added increases with the copper present ; the usual limits are 4 to 10 grams. This statement does not contradict the preceding table, as the object here is not so much to completely remove the copper as it is to ensure a good cupellation.

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\* Rose, Metallurgy of Gold, p. 402.

(2) The cupels are placed in the muffle, beginning at the back, and heated as usual. The packets containing the gold and silver are introduced by tongs and cupelled. The temperature of the muffle should be  $1050^{\circ}$  to  $1100^{\circ}$  C. (bright orange-red). The cupels are left in for 15 to 20 minutes after the buttons have become dull to get rid of all the lead, then removed from the furnace, and when cold the beads are cleaned with a stiff brush.

(3) Buttons are flattened by striking with a hammer, first in the centre, then a blow on either side to elongate. They are then annealed in the muffle by heating to redness in an iron tray or clay annealing-cup. When cold, they are passed through rolls so adjusted that one passage reduces them to a ribbon the thickness of a visiting-card (called a fillet). They are annealed again, and rolled into cornets between the thumb and finger or around a rod so that the bottom (which may contain traces of bone-ash or litharge) shall be on the outside.

The edges of the fillets should be smooth.

(4) Parting is done in a conical glass flask (see Fig. 33) or a platinum boiler (see Fig. 37). This consists of a number of small platinum cups arranged in a tray of the same metal which can be set inside of the platinum vessel (*a*). This is covered and connected with an arrangement for condensing the acid fumes. Nitric acid 1.2 sp. gr. is used first and the treatment kept up for 15 minutes at a boiling heat; this is followed by 1.3 sp. gr. nitric acid for the same time. The cornets are then washed and annealed at a high temperature, just below their melting-point.

The use of platinum boilers saves both time and acid when the operation is done on a large scale.

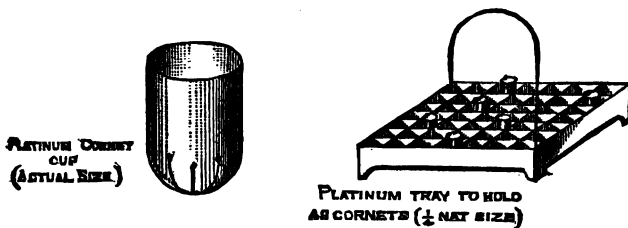
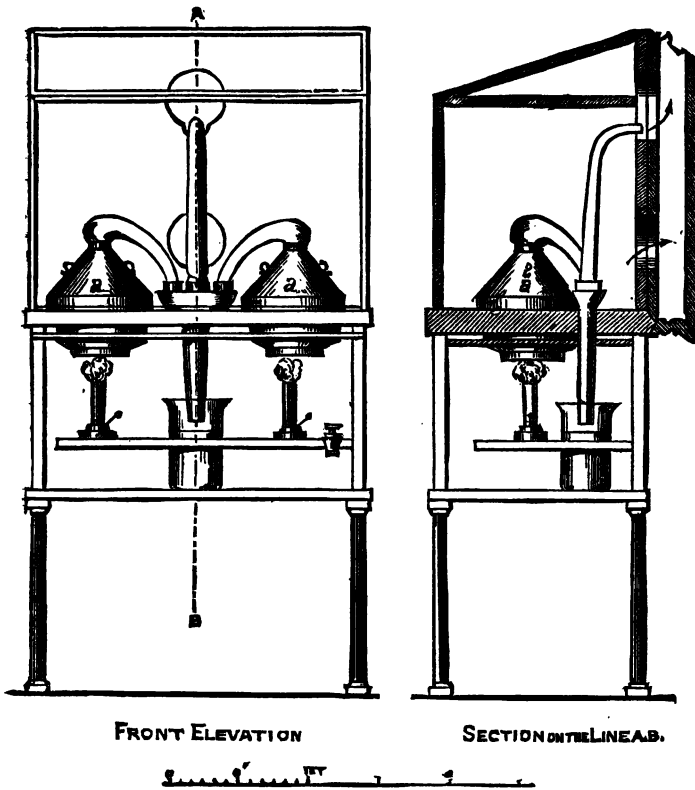


FIG. 87.

## PLATINUM APPARATUS

For Parting Gold and Silver, as used in the Royal Mint, London.

(5) The cornets are weighed on a most accurate balance. The check assays are weighed first and their mean excess or deficiency in weight applied to all the assays run with them. This correction is called "surcharge."

REMARKS.—The check assays are made up from pure gold and run with the regular assays. The pure gold is prepared as follows: \* Gold cornets are dissolved in aqua regia, the excess of acid is expelled, and alcohol and potassium chloride added to precipitate any traces of platinum. The chloride of gold is then dissolved in distilled water in the proportion of  $\frac{1}{2}$  oz. of metal to the gallon, and the solution set aside for several days to allow the silver chloride to separate. The clear liquid is then removed by a siphon, crystals of oxalic acid are added to it, and the liquid warmed gently until it becomes colorless; this requires 3 to 4 days for 10 gallons. The spongy gold so obtained is washed repeatedly with hydrochloric acid, distilled water, ammonia, and distilled water, in the order given, then melted in a clay crucible with potassium bisulphate and borax, and finally poured into a stone mould. The United States Mint uses for check assay of gold coins an alloy of gold 900, copper 75, and silver 25 parts. In the New York Assay Office the ratio of gold to silver used is 1 to 2. Nitric acid 32° B. = 1.26 sp. gr. is used for both boilings.

*Surcharge.*—The gold cornet does not contain all of the gold present in the original alloy and nothing else. Gold is lost by volatilization, by absorption in the cupel, and by solution in the acid. On the other hand, the gold always contains some silver and occluded gases. The algebraic sum of these losses and gains is called the surcharge.

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\* Rose, Metallurgy, p. 10.

The losses of gold are found to be 0.4 to 0.8 of a part in 1000, of which 82% is absorbed by the cupel, 8% dissolved by the acid, and the rest (10%) volatilized.\* The gain due to silver and occluded gases is 0.8 to 0.9 of a part in 1000. By using a balance sensitive to  $\frac{1}{100}$  of a milligram, taking every precaution, and the average of three concordant results, the fineness of gold bullion can be determined to within  $\pm 0.02$  parts in 1000.

Doré bullion is an alloy of gold and silver. (For the determination of silver see Silver Bullion.)

Impure alloys of gold with arsenic, antimony, iron, nickel, zinc, etc., require scorification.

Aluminum alloys must be treated by wet methods.

For platinum-gold alloys see Platinum (page 130).

#### SPECIAL ALLOYS OF GOLD.

1. *Auriferous Tin*.—Oxidize in muffle. Scorify with lead, 16 pts., borax glass,  $2\frac{1}{2}$  to 3 pts., and cupel button. A mixture of sodium carbonate and nitrate is sometimes added in scorifying to form sodium stannate.

2. *Auriferous Mercury*.—Distill if possible, and then scorify the residue at a *low heat* with about 10 pts. of pure lead. It is best to place the scorifier with charge in the muffle when the latter is cold, and let it heat up with the furnace.

3. *Palladium-gold*.—Alloy with 4 times its weight of silver, and part with nitric acid. The palladium dissolves with the silver.

4. *Rhodium-gold*.—Alloy with 4 parts of silver; part with nitric acid, and fuse residue with bisulphate of potash; treat with distilled water, and dry and weigh the

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\* Rose, *Metallurgy of Gold*, pp. 417-420.



gold residue from the fusion. If this is not pure, the fusion should be repeated.

5. *Iridium-gold*.—Heat with very dilute aqua regia (1:5) until the gold has dissolved; wash and weigh the iridium, which remains as a black powder. See first method under Platinum.

#### TOUCHSTONE ASSAY.

Alloys of gold can be examined for their approximate fineness by means of the *touchstone* and nitric acid. The *touchstone* is a piece of black smooth stone (basalt or slate), on which the alloy to be tested can be rubbed. The streak left is then tested with nitric acid in comparison with the streak left by an alloy of known fineness. Test-acid is sometimes employed instead of nitric acid; this is a mixture of nitric acid (sp. gr. 1.34) 98 pts., muriatic acid (sp. gr. 1.17) 2 pts., and distilled water 25 pts.

The standard alloys are called *touch-neededles*, and consist of pure gold, alloyed in various proportions with copper, silver, or both metals. They are much employed by jewelers, and can be purchased ready for use.

Where the amount of gold is small, the *touchstone* method is less accurate; but for rich alloys and preliminary work it may prove useful. A sharp eye and practice are, however, required to arrive at anything like good results.

#### PLATINUM. *Symbol*—Pt.

SOURCES.—Native platinum, either alloyed or associated with iridium, palladium, osmium, rhodium, ruthenium, iridosmium, gold, silver, copper, or iron. Platiniferous

sand. Sperrylite, ( $\text{PtAs}_2$ ) (56.5% platinum). Placer gold, some old jewelry, and other alloys often contain platinum.

**ASSAY.**—The following methods are given as the shortest accurate methods for the determination of platinum without the use of an oxyhydrogen blowpipe. When carefully conducted, they compare favorably with the long and tedious wet methods.

The material to be assayed may be conveniently divided as follows :

1st. Low-grade platinum ores, often containing considerable quantities of iridosmium.

2d. Russian ores, about 80% platinum. These ores are treated with mercury before they are sold.

3d. Platinum alloys, containing silver, gold, etc.

4th. Platinum-foil, crucibles, etc., often containing iridium, etc.

In the first case the ore must receive a preliminary treatment to concentrate the platinum ; this can be done by panning down, where the platinum is in grains, or by fusion of 2 or 4 assay tons with litharge, etc., as in the crucible assay of a low-grade gold ore, which collects the platinum in a lead button.

The chief difficulty in the assay of Russian ores is to secure an accurate sample. To obtain this melt a large quantity of the ore, 20 to 50 grams, with six times its weight of test-lead until the platinum has alloyed with the lead,—this may be done in a scorifier,—then pour, detach the slag carefully, as the resulting button is brittle, and weigh the lead-platinum alloy. Pulverize in a mortar and weigh out portions for assay. When the assay is finished, the platinum in the lead must be recovered.

*First Method,\* for Platinum, Gold, Iridium, and Iridosmium.*—Take of the lead button or platinum alloy such a quantity as shall contain about 100 milligrams of platinum, and scorify at a high heat with 40 grams of test-lead until the slag covers over, pour into a mould, and when cold hammer to detach the slag. The button should weigh about 10 grams, and will be malleable if it contains less than 5% of platinum.

Place the button in a large beaker with 200 c.c. of nitric acid 1.08 sp. gr. and heat until all action ceases; then filter through a small ashless paper, and wash the residue of lead, gold, platinum, iridium, etc., with water once or twice, transfer the paper to a porcelain capsule, dry, and then ignite in the muffle with the door open for ten minutes to oxidize the lead remaining with the gold, etc. After cooling heat to boiling with 1.08 sp. gr. nitric acid for several minutes, decant, wash thoroughly in the capsule, dry, anneal, and weigh the gold, platinum, iridium, and iridosmium.

Replace in the capsule, and warm with dilute aqua regia (1:5) for a few minutes, which readily dissolves the gold and platinum in the finely divided state in which they are left. Decant into a small beaker, ignite, and weigh the residue.

To separate iridium from iridosmium boil the residue with strong aqua regia, decant, and wash; the final residue is iridosmium.

Evaporate the filtrate containing gold and platinum just to dryness to remove chlorine and nitric acid, but do not bake as chloride of gold will be reduced, dissolve in water and a few drops of hydrochloric acid, and precipi-

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\* E. H. Miller, *School of Mines Quarterly*, Vol. XVII., pp. 26-38.

tate the gold by warming with crystals of oxalic acid for half an hour, filter, and weigh the gold,—or, better, cupel with silver (three times the weight of gold) to remove filter-ash,—part with nitric acid as usual, and weigh gold after annealing. This treatment would also remove any trace of platinum. The platinum is estimated by difference, or may be precipitated as  $(\text{NH}_4)_2\text{PtCl}_6$  after destroying the excess of oxalic acid.

If an ore contain Pt, iridosmium, Os, Ir, Pd, Ru, Rh, Ag, Au, Cu, Fe, and  $\text{SiO}_2$ , the scorification with lead removes iron and silica as well as most of the copper, and volatilizes some osmium. The first treatment with 1.08 nitric acid dissolves most of the lead, all of the silver, copper if present, palladium, and some rhodium, leaving Pb, Pt, Ir (Ru, Rh, Os?), Au, and iridosmium. The second treatment with nitric acid after ignition takes out the rest of the lead. Dilute aqua regia dissolves gold and platinum, leaving iridosmium, iridium, and some of the trifling amounts of Ru, Rh, and Os present.

*Second Method, for the Determination of Base Metal, Silver, Gold, Platinum, and Iridosmium.*—This method takes advantage of the solubility of platinum in nitric acid when alloyed with at least 12 parts of silver.

The button or platinum alloy, containing from 100 to 200 milligrams of platinum, is cupelled with sufficient silver to prevent freezing,—this should be 5 to 6 times the platinum present,—the heat should be very high at the end, and the button should be allowed to remain in the muffle for several minutes after brightening to remove lead as completely as possible, even if some silver is lost. After allowing for the silver added, the loss in weight is base metal. The button is hammered as usual, rolled out, and

the silver dissolved by boiling for several minutes with concentrated sulphuric acid; the residue is washed, annealed, and weighed; the loss minus the silver added gives the silver, the residue is gold, platinum, iridosmium, etc. To the residue twelve times its weight of silver is added and the mixture cupelled with lead; the resulting button is hammered, rolled out, and parted with nitric acid 1.16, then with 1.26 sp. gr.

One such treatment will not remove all the platinum, so that the operation must be repeated or the boiling with acid prolonged until the residue no longer diminishes in weight. Unless this precaution be observed the gold will be too high. The residue of gold, iridosmium, etc., is weighed and the loss from the previous residue determined as platinum. It is then treated with *dilute* aqua regia to dissolve the gold, and the final residue of iridosmium is obtained.

In this method no account is taken of the other metals always associated with platinum. Palladium dissolves with the silver; iridium is left with the iridosmium if the aqua regia used is dilute (1 to 5) and only heated gently; if strong aqua regia is used, the iridium will dissolve with the gold.

REMARKS.—Some very pure platinum-foil assayed by the first method gave 99.70 per cent. of platinum.

The following results were obtained on an alloy containing lead, silver, gold, platinum, iridium, and iridosmium:\*

Wet Method.—Pt, as  $(\text{NH}_4)_2\text{PtCl}_6$ , 10.53 and 10.30%; Au, by oxalic acid, 3.42 and 3.424%.

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\* E. H. Miller, S. of M. Quarterly, Vol. XVII., pp. 26-38.

First Method: Pt, 10.44 and 10.43%; Au, 3.456 and 3.45%; Ir, 0.5%; IrOs, 0.55%.

Second Method (residue not retreated):

|  | Platinum. | Gold. |
|--|-----------|-------|
| Ratio of silver to residue, 13 to 1 .. | 10.02%    | 4.88% |
| “ “ “ “ “ 19 to 1 ..                   | 10.29“    | 4.70“ |
| Iridosmium .....                       | 0.22“     |       |

These results are inserted to show the necessity for the modifications described under the second method.

Platinum if present in an ore to the extent of a small percentage of the gold and silver will not be noticed unless specially tested for, as it dissolves in the nitric acid with the silver. If present in greater quantity the button will freeze in cupellation before all the lead has been oxidized, and the bead will have a white, frosted appearance, sometimes resembling a cauliflower.

The addition of cadmium to an alloy containing platinum and silver renders the platinum insoluble in nitric acid. This fact is made use of for the determination of silver in gold bullion containing platinum.\*

### CARBON. *Symbol*—C.

(COAL.)

SOURCES.—Carbon occurs in a vast number of compounds, combined with hydrogen, oxygen, and often nitrogen, but we only give such as are of interest to the assayer:

|   | Percentage of Carbon. |
|---|-----------------------|
| Diamond, crystallized.....                    | 100 %                 |
| Graphite, nearly pure.....                    | 95 to 100 “           |
| Anthracite, volatile matter less than 7%..... | 90 to 95 “            |

\* Rose, *Metallurgy of Gold*, pp. 422 and 426.

|  | Percentage of<br>Carbon. |
|--|--------------------------|
| Semi-anthracite, volatile matter less than 10 %. | Variable                 |
| Semi-bituminous, " " " " 18 "                    | "                        |
| Bituminous, " " more " 18 "                      | "                        |
| Lignite or brown coal.....                       | "                        |
| Charcoal.....                                    | "                        |

ASSAY.—Methods for the assay of coal are all that can be given here.

The assay of a specimen of coal varies with the purpose for which the coal is to be employed. The most general tests are calorific power, specific gravity, moisture, volatile combustible (gas), fixed carbon, ash, sulphur, and in some cases phosphorus.

The calorific power of a fuel may be determined by three methods :

1st. By combustion in a calorimeter in which a known weight of water is heated by the combustion of the fuel.

2d. By calculation from the results of an elementary analysis in which C, H, N, and S, are determined.

3d. By calculation from the amount of lead reduced by one gram of the coal (Berthier's method).

The third method is the most rapid, and, though not exact for coals containing much volatile matter, is largely used.

Weigh out three portions of 1 gram each of the finely and freshly pulverized coal, and mix each with 75 grams of litharge, which should be free from red lead, place in a sand crucible and cover with 25 grams more litharge, fuse either in the crucible furnace or the muffle at a good bright heat for 20 to 30 minutes, either break or pour the crucibles, hammer and weigh the buttons as usual; aver-

age the weights of the three buttons, and multiply by 240 for an anthracite and by 268.3 for a bituminous coal. \*

The factor for pure carbon is obtained as follows: 1 gram of carbon when burned completely gives 8080 heat units, lesser calories, that is, it will raise 8080 grams of water from 0° to 1°C., and 1 gram of carbon reduces 34.5 grams of lead ( $2\text{PbO} + \text{C} = 2\text{Pb} + \text{CO}_2$ ;  $12\text{C} = 414\text{Pb}$ ), so that the weight of lead reduced multiplied by  $\frac{8080}{34.5}$  or 234.2 = the calorific power.

The factor for hydrogen, however, is  $\frac{34500}{103.5}$  or 333, so the more volatile matter the coal contains the higher must be the factor used.

To determine moisture, weigh out 1 to 2 grams of the coal in a platinum or porcelain crucible, and heat for one hour in an air-bath at a temperature of 105° to 110°C., cool in a desiccator and weigh; loss in weight is moisture.

*Volatile Combustible Matter.*—The volatile combustible is found as follows: Weigh out a fresh portion of 1 to 2 grams and heat in a platinum crucible for  $3\frac{1}{2}$  minutes over a Bunsen burner; then immediately transfer to a blast-lamp and continue heating for  $3\frac{1}{2}$  minutes more. The cover should remain on the crucible throughout. Cool and weigh; the loss is moisture, volatile combustible, and one half the sulphur present as pyrites.

*Fixed Carbon and Ash.*—After driving out the volatile combustible, etc., remove the cover from the crucible, turn it on its side, and burn out the carbon over a Bunsen burner, until nothing but a red or gray ash remains; cool

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\* Noyes, J. Am. Chem. Soc., Vol. XVII., p. 848.



and weigh; the loss in weight is fixed carbon and one half sulphur as pyrites, the residue is the ash.

*Sulphur*.—The determination of sulphur is most conveniently made as follows: Mix 1 to 2 grams of the coal with 1 gram of oxide of magnesium in which the sulphur has been accurately determined, and  $\frac{1}{2}$  gram of anhydrous carbonate of soda; place in a large platinum crucible, turn on its side, and heat at a low red heat, without the cover, for about one hour, stirring from time to time with a stout platinum wire, until the carbon has been completely oxidized. The charge should always be in a porous condition, not fused, so that the air can penetrate and oxidize the carbon and sulphur, which latter then combines with the bases present. When the carbon has been burned out, allow the crucible to cool, and mix with the white residue 1 gram of finely ground ammonium nitrate to complete the oxidation of the sulphur. Heat gently till the ammonia salts are driven out; transfer the contents of the crucible to a beaker; add about 150 c.c. of distilled water, and boil for fifteen minutes to dissolve the soluble sulphates. Filter, wash with hot water, and acidify the filtrate with hydrochloric acid. Heat to boiling, and while boiling add gradually 10 c.c. of a 10% solution of barium chloride, boil for five minutes, allow to settle, filter, and wash the precipitate of barium sulphate, first with dilute hydrochloric acid, then with hot water, until free from chlorides. Burn the precipitate in a weighed platinum crucible after moistening with a few drops of concentrated nitric acid; when white, allow to cool, and add a few drops of dilute sulphuric acid to reconvert to sulphate any barium sulphide reduced by the carbon of the filter. Ignite and weigh.

From the weight of the precipitate calculate the percentage of sulphur in the coal.

REMARKS.—Analysis of two samples of different bituminous coals gave the following results in one hundred parts:

|  |        |        |
|--|--------|--------|
| Moisture .....   | 3.310  | 0.965  |
| Volatile combustible matter + $\frac{1}{2}$ sulphur... | 27.300 | 30.111 |
| Fixed carbon + $\frac{1}{2}$ sulphur.....              | 61.965 | 61.033 |
| Ash, including phosphorus.....                         | 7.425  | 7.829  |
| Sulphur.....   | 3.863  | 1.347  |

$27.300 - \frac{3.863}{2}$ , and  $30.111 - \frac{1.347}{2}$ , give the correct amounts of volatile matter.

$61.965 - \frac{3.863}{2}$ , and  $61.033 - \frac{1.347}{2}$ , give the correct amounts of fixed carbon.

The sulphur may, however, be present either wholly or in part as sulphate, in which case it will be found in the ash, and not with the volatile matter or fixed carbon.

It is often desirable to know the amount of coke a coal will give; this is the sum of the fixed carbon and ash, or it may be determined as follows:

Weigh out enough of the coal broken up in small pieces to fill a No. 6 Hessian crucible  $\frac{3}{4}$  full; lute on the cover, leaving a hole for the escape of the gas; place in a crucible fire, and heat till no more gas escapes. Cool, weigh the coke, and examine its quality, density, etc.

For the determination of phosphorus in the ash see iron ore (page 186).



## **PART III.**

### **WET ASSAYS OR ANALYSES.**



## SILVER BULLION.

This assay can be best described under three heads:  
*a.* Preparation of solutions. *b.* Preliminary assay. *c.* Assay proper.

(*a*) PREPARATION OF SOLUTIONS.—Three solutions are required, called normal salt, decime salt, and decime silver.

The normal salt is a solution of salt 100 c.c. of which will precipitate exactly 1 gm. of pure silver.

The decime salt is a solution one tenth the strength of the normal. One c.c. will precipitate one milligram of silver. It is made by diluting one part of the normal solution with nine parts of pure water.

The decime silver is a solution of one gram of pure silver in nitric acid diluted to a litre. One c.c. of this solution will contain one milligram of pure silver. One c.c. decime silver is, therefore, equivalent to one c.c. decime salt.

“Normal” as used here has a special meaning; a normal solution usually means one containing as many grams to the litre as there are units in the molecular weight of the substance dissolved.

A large quantity of the normal salt solution is prepared and preserved in a common glass carboy, which has affixed to it a paper scale carefully graduated, indicating at any time the amount of solution left. It is made by diluting 2.07 parts of a saturated solution of salt with 97.93 parts of pure water, or until each 100 c.c. of the resulting solution contains just 0.54207 of a gram of salt, which is the amount necessary to precipitate 1 gm. of pure silver. The

amount of saturated solution required for 100 c.c. of the normal depends upon the temperature, so it is preferable to weigh out a multiple of 0.54207 grams of salt; dissolve it in water and then dilute to the proper volume. The normal solution must be well mixed, and the tubes and pipette washed out by allowing some to run through them. The solution must then be accurately standardized. For this purpose three or four solutions of silver in nitric acid are prepared, called check assays, each containing 1 gm. pure silver. The solutions are made with strong acid in glass-stoppered bottles of 8 ozs. or 250 c.c. capacity. Prepare also a temporary decime salt solution by diluting 25 c.c. of the approximate normal with 225 c.c. of water.

Three cases may arise: (1) the solution is too weak, (2) the solution is exactly right, (3) the solution is too strong.

(1) Fill the pipette with the normal salt solution and allow it to run into an empty beaker to rinse out the pipette; then run 100 c.c. into the bottle containing 1 gram of silver, shake vigorously, and allow the precipitate of silver chloride to settle in a dark place; the solution should be bright and clear; if it appears milky, shake again. Now add 1 c.c. of decime salt; if the normal solution is too weak, the addition of salt will produce a precipitate, as the silver is in excess. Continue adding decime salt as long as a precipitate is produced; then check back by adding decime silver, first 1 c.c., then  $\frac{1}{2}$  c.c. at a time.

A convenient way of recording the amount added is as follows:  $\frac{100}{-}$ ; each + sign = 1 c.c. decime salt, each

|   |   |
|---|---|
| — | + |
| — | + |
|   | + |
|   | + |
|   | + |

— sign = 1 c.c. decime silver. This means that 5 c.c. of

decime salt was added, but the last gave no precipitate, hence was not counted. The silver solution added only confirms the result that 100.4 c.c. was the correct amount, for the first silver added of course precipitated the last salt, which was in excess, the second gave no precipitate, hence the test is complete, and the strength of the solution is such that 100.4 c.c. = 1 gm. Ag.

(2) If the strength of the solution is right, the first salt will give no precipitate, neither will the second silver.

$\frac{100}{\equiv | +}$ ; 100 c.c. = 1 gm. Ag, or the solution is exactly normal.

(3) If the solution is too strong, the addition of salt will give no precipitate; then silver is added until it fails to give a precipitate.

$$\frac{100}{\equiv | +}$$

means that it requires 1.002 grams of silver to precipitate the salt in 100 c.c. of the solution, or  $99.8 + \text{c.c.} = 1 \text{ gm. Ag.}$  For every 99.8 c.c. of solution 0.2 c.c. of water must be added to make it normal. In the first case to make the solution normal we must add salt equivalent to  $\frac{1}{100}$  of 0.54207 grams for every 100.4 c.c. of solution.

Suppose we have six litres of solution left, the excess of water ( $x$ ) can be calculated as follows:  $100.4 : 0.4 :: 6000 : x$ ; then  $100 \text{ c.c.} : x \text{ c.c.} :: 0.54207 : y$  ( $y$  = grams of salt to add).

(b) PRELIMINARY ASSAY.—This has been described in Part II. It is necessary to make a preliminary assay in order to determine the weight of the alloy that will precipitate almost exactly the chlorine in the 100 c.c. of salt. Unless this is done, the assay becomes tedious and inaccurate, and does not conform with the practice of the



mints and assay offices. If the alloy contains but little copper, the preliminary assay may be made with sufficient accuracy by Volhard's method\* of titrating with a standard solution of ammonium sulphocyanide, using ferric chloride as an indicator.

(c) ASSAY PROPER.—Take such weight of the alloy as contains one gram of silver; if the corrected approximate fineness is 910,  $910 : 1000 :: 1 : x$  ( $x = 1.099$ , which is the weight to be taken). This is weighed out carefully, dissolved by 10 c.c. of nitric acid 1.26 sp. gr., in an 8-oz. bottle provided with a good ground-glass stopper, by heating gently on a sand-bath. Blow out the fumes and allow to cool, then run in 100 c.c. of normal salt and proceed as in standardizing.

Suppose the result is as follows:  $\frac{100}{\begin{array}{c} + \\ + \\ + \\ + \\ + \end{array}}$ ; hence 100.3 c.c.

salt solution were used. If the solution is exactly normal, we have  $1.099 : 100.3 :: 1000 : x$  ( $x = \text{fineness}$ ). If the solution is not normal, another correction must be made before using this proportion. (See examples in Part V.)

*Apparatus Employed.*—Fig. 38.—The carboy should hold about 60 litres or 15 to 16 gallons, and have a paper scale affixed to it, graduated by adding successively a known number of litres of water until the carboy is filled, and marking after each addition the height of the liquid. *B* and *V* are parts of a valve. *B* is the cover of glass through which the tubes pass, fitted by a cork. *V* is a neck of sheet iron four inches deep. The valve is

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\* See Determination of Arsenic.

closed with mercury to about one-third of its height. An enlarged section of the valve is shown at *Y*. The tube *T* and the siphon *S* reach nearly to the bottom of the carboy. The former admits air, and as none can pass out evaporation is prevented. The siphon is jointed with

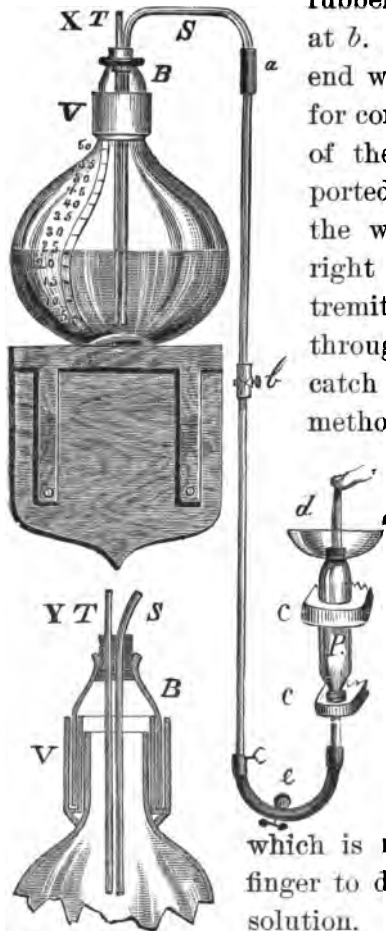


FIG. 38.

rubber at *a*, and has a stop-cock at *b*. It is furnished at the lower end with a piece of rubber tubing for connecting with the lower part of the pipette *P*, which is supported by brackets *cc* affixed to the wall of the room or an upright standard. The upper extremity of the pipette *P* passes through a vessel *d*, designed to catch the liquid running over. The method of using the apparatus is to attach the tube to the pipette, open the pinch-cock *e*, and allow the normal solution to flow upwards into the pipette until the latter overflows. Stop the flow, close with the finger, remove the rubber tube, and wipe off any of the solution adhering to the outside of the pipette, which is now ready on removing the finger to deliver 100 c.c. of the normal solution. Fig. 39 shows a very convenient apparatus for holding the glass bottle which receives the liquid from the pipette and for catch-

ing the liquid from the pipette and for catch-

ing the drippings. *C* is a cylinder of tin plate to receive the assay-bottle. *E* is a sponge enveloped in linen, forced into a tube of tin, terminated above by a cup and open below, so that the liquid may run into the vessel *B*, on which the tube is soldered. The whole apparatus is affixed to a sheet of tin plate, movable in two slots *R R*. The extent of movement is determined by two stops *t t*, so placed that when the base of the apparatus abuts against one of them the pipette will be in contact with the

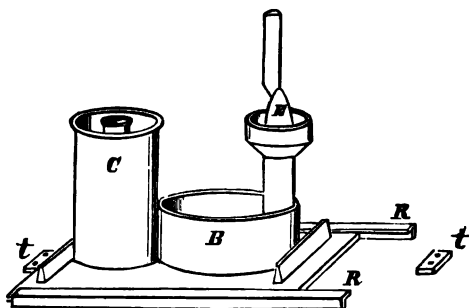


FIG. 39.

sponge. When it strikes the other, it will be directly over the centre of the neck of the bottle.

**REMARKS.**—The precipitated chloride of silver must be exposed to the light as little as possible. This may be prevented by windows of yellow glass, which excludes chemical rays. If the bullion treated contains mercury, sunlight will not blacken the combined chlorides; the mercury may be held in solution by adding 10 gms. of sodium acetate and a little free acetic acid. Test for the presence of mercury by standing the bottle in the light. The temperature of the normal solution should remain the same as that at which it was standardized; the most convenient temperature is 68° F. The solution should be

made and kept in a separate room, the temperature of which can be regulated.

Despite all precautions the normal solution will become stronger in time by the evaporation of water. This will demand correction, and in practice it is customary to take a certain weight of pure silver and subject it to the same operation as in the regular assay. The latter is corrected according to the indications of the proof assay. 1.004 grams of silver is a convenient weight to take.

The presence of sulphide of silver or antimony, lead, and tin sometimes interferes in making the silver bullion assay. The first two may be removed by boiling with stronger acid. For the latter a little nitre and sulphuric acid will make a clear solution.

#### LEAD. *Symbol*—Pb.

SOURCES.—See page 63.

ASSAY. (1) *Gravimetric Method* (in which the lead is weighed as sulphate,  $\text{PbSO}_4$ ).—Dissolve 1 gram of finely pulverized ore or  $\frac{1}{2}$  gram of an alloy in 15 c.c. of strong nitric acid in a covered casserole by the aid of heat; when the solution is complete and the sulphur oxidized, add 10 c.c. of sulphuric acid, 1.41 sp. gr., and evaporate to fumes of sulphuric anhydride,  $\text{SO}_3$ . If the solution bumps near the end, keep the contents of the casserole revolving while holding in the hand over a flame. Allow to cool, and add carefully, 100 c.c. of water; break up the residue, and heat to boiling for a few minutes to dissolve any basic ferric sulphate that may be formed by the evaporation. Allow to cool, and then decant through a small filter, leaving as much as possible of the residue of  $\text{PbSO}_4$ ,  $\text{CaSO}_4$ ,  $\text{SiO}_2$ , etc., in the casserole. Wash this residue thoroughly with 1%

sulphuric acid, then with alcohol; pour the washings through the filter each time.

Dissolve the lead sulphate ( $\text{PbSO}_4$ ) from the residue in the casserole in a strong hot solution of ammonium acetate slightly acid with acetic acid; pour this solution through the filter into a clean beaker; repeat this till all the lead sulphate is dissolved, then wash the contents of the casserole onto the filter with hot water. Acidify the filtrate with sulphuric acid and filter the  $\text{PbSO}_4$  on a small paper, washing as before with dilute sulphuric acid and with alcohol. Dry the filter and lead sulphate, separate the precipitate from the paper, and burn the paper with a few drops of strong nitric acid in a weighed porcelain crucible. If any lead has been reduced by the carbon of the paper, dissolve this in nitric acid, add sulphuric acid (a few drops), and evaporate to dryness. Now add the remainder of the precipitate, ignite, and when cold, weigh; the increased weight of the crucible and cover minus the weight of the filter-ash is  $\text{PbSO}_4$ , from which the percentage of lead is calculated.

When filter-paper is used which has been treated with hydrofluoric acid, the ash is so small that it may be neglected. The ignition of the lead sulphate may be done in a platinum crucible without injury to the crucible if care is taken to prevent the reduction of any lead.

(2) *Volumetric Method, Alexander's*.<sup>\*</sup> — This method is based on the fact that ammonium molybdate when added to a hot solution of lead acetate will give a precipitate of molybdate of lead ( $\text{PbMoO}_4$ ) which is insoluble in acetic acid. Any excess of ammonium molybdate

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<sup>\*</sup> Engineering and Mining Journal, April 1893, p. 298.

will give a yellow color with a freshly prepared solution of tannin.

*Solutions Required.*—A standard solution of ammonium molybdate containing about 9 grams per litre. If the solution is not clear, add a few drops of ammonia.

*Indicator.*—A freshly prepared solution of 1 part tannin in 300 parts of water.

*Standardizing.*—Weigh out 300 milligrams of pure dry sulphate of lead, dissolve it in hot ammonium acetate, acidify with acetic acid, and dilute with water to 250 c.c., heat to boiling, and run in ammonium molybdate solution from a graduated burette till the lead is all precipitated as white  $\text{PbMoO}_4$ . This is ascertained by placing drops of the indicator on a white surface, such as a porcelain plate, and to them adding a drop of the solution tested after each addition of ammonium molybdate: as long as the lead is in excess no coloration is produced; when the end is reached, a very slight excess of ammonium molybdate gives a yellow color with the tannin.

In titrating it is essential to stir the solution very thoroughly, and when near the end to wait a few seconds before trying the drop test.

*Assay.*—The ore is treated as described under the gravimetric method until the lead is precipitated as sulphate; this is washed twice with hot dilute sulphuric acid, then once with water, and dissolved as before in hot ammonium acetate; this solution is acidified with acetic acid diluted to 250 c.c. with hot water, and heated to boiling. The solution is now ready for titration, which is done as in standardizing. The number of c.c. used times the strength of the solution in terms of lead divided by the weight taken and multiplied by 100 gives the per cent of lead.

If the titration is delayed and the solution becomes cold, it should be heated again almost to boiling before the end is reached.

REMARKS.—Three lead ores gave the following results by the methods described:

|                        | Gravimetric. | Volumetric. |
|------------------------|--------------|-------------|
| Galena... ..           | 75.10        | 74.98       |
| Galena and pyrites.... | 33.74        | 33.80       |
| Cerussite....          | 38.48        | 38.20       |

For results by fire assay on these ores see page 66.

### ZINC. *Symbol*—Zn.

SOURCES.—The principal ores of zinc are:

|   |               |
|---|---------------|
| Blende (sphalerite), sulphide ( $\text{ZnS}$ ) when pure                  | = 67.7 % zinc |
| Smithsonite, carbonate ( $\text{ZnCO}_3$ )... “ “                         | = 52.0 “ “    |
| Calamine, silicate ( $\text{Zn}_2\text{SiO}_4 + \text{H}_2\text{O}$ ) “ “ | = 53.8 “ “    |
| Willemite, silicate ( $\text{ZnSiO}_3$ )..... “ “                         | = 58.3 “ “    |
| Zincite, oxide ( $\text{ZnO}$ ) ... .. “ “                                | = 80.26 “ “   |

The last two occur associated with Franklinite.

The first three are found alone or associated with the ores of other metals; this being especially true of the sulphide, which often contains silver and is found with galena.

ASSAY. (a) *Gravimetric*.—The detailed methods are long and tedious and do not come within the scope of this book; the reader will find them described in Cairns' Quantitative Analysis, Crooke's Select Methods, Fresenius' Quantitative Analysis, etc. Only a brief outline of the quantitative practice is given as follows:

Obtain the zinc, from about 1 gram of ore, in solution as chloride as is described under the volumetric method; filter off the insoluble residue, and saturate the hydrochloric acid solution with hydrogen sulphide ( $H_2S$ ) to precipitate any lead, copper, cadmium, arsenic, antimony, or bismuth, etc., which may be present; filter; in the filtrate oxidize the iron by adding a few crystals of potassium chlorate and boiling; nearly neutralize with sodium carbonate until the solution becomes dark red in color, but remains clear; then add sodium acetate until the color no longer darkens; dilute to at least 500 c.c. and boil for three minutes, filter, and wash with hot water. The precipitate will contain iron, aluminum, phosphorus, etc.; the filtrate zinc, manganese if present, lime, magnesia, etc. To the filtrate add a few c.c. of acetic acid, then bromine, and warm until the manganese is completely precipitated as hydrated  $MnO_2$ ; filter and wash; heat the filtrate to boiling, add 5 grams of ammonium chloride, and while hot saturate with hydrogen sulphide; filter out the zinc sulphide and wash with water containing hydrogen sulphide; dissolve in nitric acid, evaporate off the excess, dilute, and add a decided excess of microcosmic salt; heat to boiling and add ammonia until faintly alkaline; heat on water-bath until the precipitate becomes crystalline; filter when cold, wash, dry, separate from paper, burn the paper with nitric acid in a weighed porcelain crucible, then add precipitate, and ignite very gently; weigh zinc pyrophosphate ( $Zn_2P_2O_7$ ), and calculate the percentage of zinc. (See table of factors, Part V.)

(b) *Volumetric*.—The following methods will be found sufficiently accurate for commercial use:

*Principle*.—Zinc is precipitated as ferrocyanide by a



standard solution of potassium ferrocyanide ( $K_4FeCy_6$ ) in a weak hydrochloric acid solution.\* The end reaction is a brown color with uranium acetate ( $UO_2(C_2H_3O_2)_2$ ). Cadmium, copper, iron, manganese, nickel, and cobalt interfere, and must therefore be removed before titration.

*Solutions.*—A standard solution of potassium ferrocyanide, made by dissolving 43.2 grams of the salt,  $K_4FeCy_6 \cdot 3H_2O$ , in cold water and diluting to one litre; 1 c.c. will be equivalent to about 10 mgs. of zinc.

A saturated aqueous solution of uranium acetate.

A saturated solution of chlorate of potash in strong nitric acid.

A solution of ammonium chloride containing 10 grams to the litre.

*Standardizing.*—As the end reaction in this method is not extremely delicate in the presence of hydrochloric acid, it is necessary to make allowance for the amount of the standard solution in excess, necessary to turn the indicator; this amount is then subtracted from the burette-reading in every case. To determine this decide upon the bulk and degree of acidity to be used, as the allowance is affected by both. For ores which do not contain lead, 200 c.c. containing 2 c.c. of HCl 1.2 sp. gr. will give good results. If lead is present, more HCl must be used to prevent the precipitation of lead ferrocyanide. Place the amount of water and acid decided on in a beaker and warm to  $80^\circ C$ .; run in ferrocyanide drop by drop until the indicator is affected: this is determined by placing drops of uranium acetate on a white plate and bringing a drop of the solution in contact with them; the first decided brown shows the end.

To standardize the ferrocyanide solution weigh out two

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\* The composition of the precipitate is probably  $Zn_2K_2Fe_2Cy_{12}$ .

portions of about 0.2 of a gram each of pure zinc (of course note the exact weights), dissolve these in beakers in a little concentrated HCl with aid of heat, dilute slightly and neutralize with sodium carbonate, then add the amount of HCl decided on (2 c.c.), dilute to the standard bulk (200 c.c.), heat almost to boiling, and run in ferrocyanide until the indicator is affected. Subtract from the amount used what was found to be necessary to turn the indicator, then divide the weight of zinc by the result. Standards should agree to the fifth decimal place.

(1) *Method for Ores Containing Iron, Copper, Cadmium, and Small Amounts of Manganese, such as Blende, Calamine, Smithsonite, etc.*—Take exactly one gram of the finely pulverized ore and treat with 25 c.c. of the chlorate solution in a casserole. Warm gently at first and continue heating till all greenish fumes have disappeared, cover, and evaporate to dryness, but do not bake; this treatment throws out the manganese as  $MnO_2$ . When cold add 7 grams  $NH_4Cl$ , 15 c.c. strong ammonia, and 25 c.c. of hot water, boil for a few minutes, and break up any masses that adhere to the sides of the casserole, filter into a flask or beaker, and wash with  $NH_4Cl$  solution, to which a few drops of ammonia have been added. If a large precipitate of ferric hydrate, etc., is formed it will carry down zinc with it; in this case transfer the precipitate, without the paper, back into the casserole, and treat as before with chlorate solution,  $NH_4Cl$  and  $NH_4OH$ , filter, and combine the two filtrates.

If this filtrate is blue, copper is probably present; it is most conveniently removed by acidifying the solution with sulphuric acid, then shaking with aluminum foil until the copper is precipitated. If the ammoniacal solution shows

no blue color, of course this treatment is omitted. If cadmium is present, it must be removed by  $H_2S$  in a hydrochloric acid solution, which will remove copper at the same time. Nickel and cobalt are not likely to be found in a zinc ore; if present, the zinc must be precipitated in a hot acetic acid solution as sulphide. (See gravimetric scheme, p. 153.)

The solution is now brought to the standard condition as to bulk and acidity. Methyl orange or some other indicator may be used here, though the color is liable to complicate the end reaction. The solution is heated and titrated as in standardizing. When the amount of zinc in the ore is not approximately known, it is best to titrate about half the solution, first running in 1 c.c. at a time, so as to get an idea of the amount of ferrocyanide required; then put in the second half, run up almost to the required amount, and finish by adding ferrocyanide drop by drop.

Some ores, such as Willemite, which is commonly found associated with Franklinite, will not yield all their zinc when treated as described. In some cases a treatment with concentrated hydrochloric acid or aqua regia previous to the chlorate treatment will yield fairly accurate results. It is necessary to remove the excess of hydrochloric acid by evaporation before adding the oxidizing mixture ( $HNO_3$  and  $KClO_3$ ) to give complete precipitation of the manganese as  $MnO_2$ .

(2) *Method for Franklinite or Ores Rich in Manganese.*—Fuse 1 gram of the ore, ground to an impalpable powder, with 10 grams of mixed carbonates of potassium and sodium, and  $\frac{1}{2}$  gram of sodium nitrate for one hour, dissolve the fusion in hydrochloric acid, boil till the acid has become concentrated, add a few crystals of  $KClO_3$  and

boil to oxidize the iron completely and to expel the excess of acid ; dilute to about 100 c.c., cool, nearly neutralize with a saturated solution of sodium carbonate, then add barium carbonate suspended in water, and shake till the precipitate curdles, which marks the complete precipitation of the iron. This is best done in a flask. Filter immediately and wash with cold water. To the filtrate add 10 c.c. of sodium acetate, 5 c.c. of acetic acid, and a few c.c. of bromine, warm under a hood until all the manganese is precipitated as hydrated  $\text{MnO}_2$ , and the excess of bromine expelled ; filter out the  $\text{MnO}_2$ , wash with water, evaporate the filtrate to about 150 c.c., neutralize the acids ( $\text{HBr}$  and  $\text{HC}_2\text{H}_3\text{O}_2$ ) with carbonate of soda, add the regular excess of  $\text{HCl}$ , etc.; and titrate as before.

The barium carbonate for this method should be specially prepared as follows :\* Select a c. p. carbonate of barium which is free from ammonia salts, such as Merck's; suspend this in water, and heat for several hours with 2 to 3 per cent. of barium chloride ; this converts any alkaline carbonates, which would precipitate zinc, to chlorides. The slight excess of barium chloride does not interfere. When this precaution is not taken, the iron precipitate may contain up to 1 per cent. of zinc.

REMARKS.—Chloride of platinum acidified with hydrochloric acid has been used as an indicator.† This gives a bright emerald-green with a hot solution containing potassium ferrocyanide. The color takes a few seconds to develop, but is less affected by a variation of the excess of  $\text{HCl}$  than uranium acetate. It will not, however, work with a cold solution.

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\* Stone, J. Am. Chem. Soc., Vol. XVII., p. 473.

† E. H. Miller, J. Am. Chem. Soc., Vol. XVIII.

No oxidizing agent must be present when the solution is titrated. If present it can be destroyed by adding a few c.c. of sulphurous acid and then boiling out the excess.

### BISMUTH. *Symbol*—Bi.

SOURCES.—See page 69.

ASSAY. *Determination of Bismuth in a Lead Alloy.*  
—Dissolve 1 to 3 grams of the alloy in dilute nitric acid in a casserole, add 5 c.c. of concentrated sulphuric acid previously diluted with water, evaporate to fumes of  $\text{SO}_3$ , cool, add water and heat to boiling, and filter out the insoluble  $\text{PbSO}_4$ , Au, etc. If silver is present, add a little salt solution before filtering. Warm the filtrate and saturate with sulphuretted hydrogen, filter and wash with  $\text{H}_2\text{S}$  water. Place the precipitate in a beaker and digest hot with yellow ammonium sulphide to dissolve sulphides of arsenic, antimony, tin, etc.; filter, dissolve the residue of sulphides of bismuth, copper, etc., in nitric acid, make alkaline with ammonia and add a slight excess of ammonium carbonate solution, boil for 15 minutes and allow to stand till the bismuth carbonate settles; filter, wash, and weigh as  $\text{Bi}_2\text{O}_3$ .

*Determination of Bismuth in a Copper Matte or Alloy.*  
—To the nitric acid solution of bismuth and copper add ammonia in excess and ammonium carbonate as already described. The precipitate may contain copper and must be redissolved in nitric acid and reprecipitated by ammonia and ammonium carbonate. Precipitate the bismuth in a moderately dilute solution.

COPPER. *Symbol*—Cu.

**SOURCES.**—Besides the ores given in Part II., page 82, slags, mattes, pig copper, etc., are frequently to be assayed for copper.

**ASSAY.**—Three methods are given :

1. Electrolytic.
2. Volumetric, “iodide” method.
3. Volumetric, “cyanide” method.

(1) *Electrolytic.*—Weigh out such quantity of ore or matte as shall contain 100 to 150 milligrams of copper. Treat in a small casserole covered by a watch-glass with a mixture of 20 c.c. concentrated nitric acid, 5 c.c. conc. HCl, 5 c.c. conc.  $\text{H}_2\text{SO}_4$ , on a hot plate till the copper has completely dissolved, then evaporate to copious fumes of  $\text{SO}_3$ . Allow to cool, and when cold add 50 c.c. of water, heat to boiling, and boil for several minutes to dissolve all the copper sulphate. Filter out the silica, etc., wash free from copper with hot water, run the filtrate and washings into a No. 1 beaker, nearly neutralize the excess of sulphuric acid with ammonia, then add 2 to 3 c.c. of conc.  $\text{HNO}_3$ .

The copper is best deposited on a platinum electrode, cylindrical in shape, having an area of at least 10 square inches; the cathode, must be thoroughly cleaned with nitric acid, washed with alcohol, heated to redness, and when cold weighed. It is then connected with the negative pole of the battery or other source of current. The anode may be a similar smaller platinum electrode or a piece of stout platinum wire bent in a spiral; this is placed inside the cathode, but not in contact with it, and connected with the positive pole. The beaker containing the copper solu-

tion is placed on blocks under the electrodes so that they reach nearly to the bottom.

The current must have a voltage of at least 1.24 to decompose  $\text{CuSO}_4$ . The ampères determine the rate of deposit; from 0.1 to 0.25 ampère will give good results in most cases. If the copper is deposited too fast, it will not adhere firmly to the platinum. The most convenient source of current is a storage-battery, but either Edison-Laland or gravity cells will work very satisfactorily.

It is usual to run a number of copper determinations at once. They are connected with the current in the afternoon and will be finished the next morning. When Edison-Laland cells are used, type S, put on one more cell than there are solutions to be electrolyzed.

The copper should be deposited in a firm red adherent coating. The current must be continued till the solution gives no blue with ammonia. If nickel is present, test by  $\text{H}_2\text{S}$  in an acid solution. When the copper has all been deposited, remove the blocks which support the beaker and immediately wash off the cathode with distilled water. The current must not be stopped while the cathode is in the solution, or the nitric acid would redissolve copper. Disconnect the cathode and rinse with alcohol, dry at a gentle heat so that the copper will not oxidize, allow to cool for five minutes, and weigh. The increase in weight is copper.

Nitric acid is used to prevent the deposition of small quantities of arsenic and antimony. If the deposition of copper takes a very long time, or if the current is strong, the excess of nitric acid will be reduced by the current and must be replaced. The cathode should be removed as soon as possible after all the copper is down, as zinc if

present, as well as some other metals, will begin to deposit after the copper.

A platinum dish or large crucible can be used as a cathode. In this case the solution must be removed by a siphon before interrupting the current.

*Interfering Metals.*—Traces of arsenic, antimony, and bismuth do not interfere with the method as described. Arsenic or antimony in considerable quantity must be removed either by roasting or by wet methods.

Bismuth must be separated by ammonium carbonate, as described under Bismuth.

Lead and manganese go to the anode as peroxides ( $\text{PbO}_2$  and  $\text{MnO}_2$ ). They do not interfere except when a dish is used. In this case they may drop to the bottom and become attached with the deposited copper.

Nickel or cobalt will not deposit in a nitric acid solution.

Zinc may come down after the copper. This is more likely to happen if the current is strong.

Silicates containing copper often require a fusion with alkaline carbonate previous to the treatment described.

With ores containing much iron it is very difficult to get the last traces of copper deposited. When but little copper is left in the solution disconnect from the current and precipitate the iron with a large excess of ammonia, filter, acidify the filtrate with nitric acid, and continue the electrolysis.

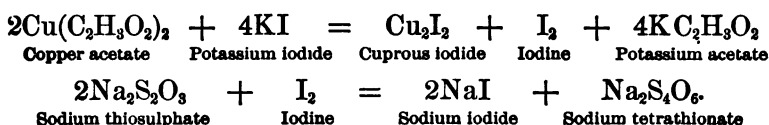
*Pig Copper.*—To obtain an average sample (see p. 51) weigh out 10 grams of fine borings, dissolve them in a flask with 50 c.c. of strong nitric acid, add a few drops of salt solution to precipitate silver, filter and wash with hot water. When cold weigh the filtrate and from it weigh



out portions for electrolysis. Examine the residue for copper.

(2) *Iodide Method*.—This method is based on the fact that potassium iodide precipitates all the copper from an acetic acid solution as cuprous iodide ( $\text{Cu}_2\text{I}_2$ ), liberating at the same time iodine. The iodine is measured by sodium thiosulphate “hypo,” using starch-paste as an indicator.

*Reactions.*—



*Solutions*.—Standard solution, containing about 19.6 grams crystallized sodium thiosulphate ( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ), per litre.

Starch solution, made by mixing 1 gram of starch to a thin paste with cold water, and then pouring into 100 c.c. of boiling water and boiling for several minutes. This solution does not keep and must be made up every few days; it must be used cold and in about the same quantity each time.

A saturated solution of zinc acetate.

*Standardizing*.\*—Weigh out two portions of fine copper-foil of about 200 milligrams each, dissolve in flasks or covered beakers in 10 c.c. of nitric acid 1.2 sp. gr., boil thoroughly till all red fumes are expelled, remove the flame, add 20 c.c. of zinc acetate solution, boil for a minute, allow to cool, and dilute with water to about 50 c.c.; add 5 grams of crystals of potassium iodide, stir till they have dissolved, and run in “hypo” solution until the

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\* Copper Assay by the Iodide Method, A. W. Low, J. Am. Chem. Soc., Vol. XVIII., p. 468.

brown color due to the liberated iodine has become weak ; add 1 to 2 c.c. of starch solution, and continue the titration until the blue color has entirely disappeared. When near the end run in the "hypo" drop by drop, stirring thoroughly between each addition. From the number of c.c. used calculate the standard of the hypo solution, which should be approximately 1 c.c. = 0.005 grams Cu.

*Assay.*—Treat 1 to 3 grams of ore or 0.5 grams of matte with 20 c.c. of concentrated nitric acid, 5 c.c. concentrated hydrochloric acid, and 5 c.c. concentrated sulphuric acid in a small casserole or flask, heat gently and evaporate to fumes of  $\text{SO}_3$  ; allow to cool ; add 20 c.c. of water and heat to boiling till all the copper is in solution. Filter out the  $\text{SiO}_2$ ,  $\text{PbSO}_4$ , etc., allowing the filtrate to run into a small beaker. Wash the casserole and filter with hot water, keeping the volume of the filtrate and washings down to about 50 c.c. if possible. Place in the beaker two pieces of sheet aluminum  $\frac{1}{8}$  inch thick and  $1\frac{1}{2}$  inches square, whose corners are bent up so that a large surface may be acted on ; add 5 c.c. of strong sulphuric acid ; cover the beaker and heat to boiling for about ten minutes or until all the copper is precipitated. Pour the solution and deposited copper into a clean beaker and allow to settle, then decant through a filter and wash with hot water ; pour the washings through the filter, but leave as much as possible of the copper in the beaker. Place the beaker under the funnel. To the beaker containing the aluminum, to which some copper may adhere, add 6 c.c.  $\text{HNO}_3$  1.2 sp. gr., and warm till all the copper has dissolved. Pour this solution through the filter into the beaker underneath containing most of the precipitated copper. Heat the beaker till all the copper has dissolved, then add half a gram of potassium

chlorate and boil for a few minutes to oxidize any arsenic precipitated with the copper. Now replace the beaker under the funnel and wash the filter and beaker containing the aluminum into it with a little hot water. We now have all the copper in the beaker as nitrate; to this add 20 c.c. of zinc acetate solution and proceed as in standardizing.

(3) *Cyanide Method*.—This method is based on the decolorization of an ammoniacal solution of copper nitrate by potassium cyanide.

The reactions are complicated, as different organic compounds are formed under varying conditions. For this reason it is essential to the accuracy of the method that the bulk of solution, the temperature, the excess of ammonia, the quantity of ammonium salts, and approximately the amount of copper in solution shall be the same as in standardizing.

*Solution for Titration*.—Made by dissolving 22 grams of pure potassium cyanide in water and diluting to 1 litre.

*Standardizing*.—Weigh out portions of pure copper of about 200 milligrams each, dissolve in 10 c.c. 1.2 sp. gr. nitric acid in a beaker capable of holding 200 c.c., boil out the fumes, dilute with cold water to about 80 c.c., add caustic soda solution till a slight permanent precipitate forms, then add 6 c.c. of strong ammonia sp. gr. 0.9. Run in cyanide from a graduated burette till the blue color is very faint, then dilute the solution to 150 c.c., and continue adding cyanide a few drops at a time until the blue color has disappeared.

The bulk of the solution should be 150 c.c. at the end in every case. To obtain this the operation is continued until but a few drops more are required, then diluted to

the correct volume. The beakers should be marked at the point they hold 150 c.c.

*Assay.*—Dissolve the matte, ore, or bullion as has already been described ; precipitate the copper by aluminum, zinc, or as sulphide by  $H_2S$  ; dissolve in nitric acid, and proceed as in standardizing.

This method will yield very accurate results when the operator is familiar with the end reaction and keeps the conditions the same in each case. The titration can be done in the presence of iron, but is not as accurate ; if it is performed in this way, standardize with a solution containing the same amount of iron. Do not attempt to filter off the precipitated ferric hydrate, as it holds copper which cannot be washed out. A better plan is to titrate until near the end, then filter, bring the filtrate to the proper volume, and finish the titration with the clear solution.

REMARKS.—A copper matte assayed by the three methods described gave the following results :

|                   | 1st.  | 2d.   | Average. |
|-------------------|-------|-------|----------|
| Electrolytic..... | 53.89 | 53.93 | 53.91    |
| Iodide.....       | 53.97 | 53.99 | 53.98    |
| Cyanide.....      | 54.14 | 54.08 | 54.11    |

When copper ores, mattes, etc., are bought by smelters on the results of wet assays, it is customary to deduct 1.3% for loss of copper in smelting, and call the result copper by American fire-assay.

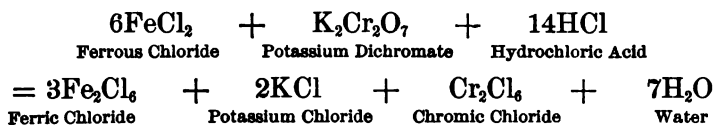
IRON. *Symbol*—Fe.

SOURCES.—See page 75.

## ASSAY.

*Iron Ores.*—The determination of iron is made by oxidizing the ferrous iron from a known weight of ore with a standard solution of either potassium permanganate or potassium dichromate. There are many modifications of these methods, but the two given are the most rapid and when properly used are extremely accurate.

(1) *Dichromate Titration* (Penny's Method): This method is based on the following reaction :



Which takes place very rapidly at the ordinary temperature. The end reaction is the absence of blue with potassium ferricyanide.

*Solutions.*—N/60 potassium dichromate, made by dissolving 4.9 grams of the pure salt and diluting to one litre. This solution keeps very well, so that frequent standardizing is unnecessary. As an indicator, a freshly prepared very dilute solution of potassium ferricyanide. Stannous chloride solution, containing 1 lb.  $\text{SnCl}_2 \cdot 3\text{H}_2\text{O}$ , 1 lb. conc. HCl in two litres. A saturated solution of mercuric chloride.

*Standardizing.*—The solution of  $\text{K}_2\text{Cr}_2\text{O}_7$  will be found to be very close to the theoretical strength, 1 c.c. = 0.0056 gms. Fe, but it should be standardized against iron wire of known purity, as follows : Dissolve two portions of flower

wire, from which any rust or dirt has been removed by emery-paper, of about 100 milligrams each, in 20 c.c. of HCl, 1.1 sp. gr. in small beakers; when solution is complete add stannous chloride drop by drop to the hot solution until it becomes colorless. Avoid an excess. Dilute to 50 c.c. and add at once 5 c.c. of mercuric chloride and stir. A white silky precipitate of  $\text{Hg}_2\text{Cl}_2$  should be formed. If there is no precipitate, the iron cannot all have been reduced. If the precipitate is gray or black, mercury has been reduced owing to the presence of too large an excess of  $\text{SnCl}_2$ . If a silky white precipitate is formed, transfer to a larger beaker, dilute to about 300 c.c., and proceed at once with the titration; if a silky precipitate is not formed, start another portion.

Place a number of drops of indicator on a white plate, run dichromate into the ferrous chloride solution until it becomes greenish, then test a drop by placing it in contact with one of the drops of ferricyanide on the plate. Continue the addition of  $\text{K}_2\text{Cr}_2\text{O}_7$  until a drop of the solution gives no blue precipitate or color with the indicator, showing that all the iron has been oxidized to ferric chloride.

*Assay.*—Weigh out 50 times the standard of the dichromate solution in terms of iron. If the solution is 1 c.c. = 0.0056 Fe, weigh out 0.280 gram in a small beaker; to this add 2 c.c. of stannous chloride and 20 c.c. of HCl, 1.1 sp. gr., and heat till the residue is white. The solution should be yellow. If it is colorless, add a little  $\text{K}_2\text{Cr}_2\text{O}_7$  or  $\text{K}_2\text{Mn}_2\text{O}_8$  until it becomes yellow, then reduce by adding  $\text{SnCl}_2$  drop by drop and proceed as in standardizing. Each c.c. of  $\text{K}_2\text{Cr}_2\text{O}_7$  used = 2% Fe without any calculation. Some ores, particularly magnetites, will not yield all their

iron by this treatment when they contain dark silicates such as pyroxene, etc. If the iron as silicate as well as that present as oxide is to be determined, fuse the ore with mixed carbonates of potassium and sodium and sodium nitrate ( $\text{NaNO}_3$ ), dissolve in  $\text{HCl}$ , filter and precipitate the iron as  $\text{Fe}_2(\text{OH})_6$  with an excess of ammonia. Dissolve this precipitate in  $\text{HCl}$  and proceed as described. If an attempt is made to reduce the iron by  $\text{SnCl}_2$  and then add  $\text{HgCl}_2$  in the presence of the alkaline salts from the fusion, a black precipitate of mercury is usually the result.

(2) *Permanganate Titration, Zimmermann-Reinhart Method.*\*—The oxidation of ferrous iron by potassium permanganate is best done in a sulphuric acid solution; but as sulphuric acid is a poor solvent for iron ores, this requires a precipitation of the iron as hydrate, then a solution by sulphuric acid, then reduction by amalgamated zinc, etc. Hydrochloric acid is liable to be oxidized by the permanganate, giving chlorine which may escape from the solution without oxidizing the equivalent quantity of iron, or make the end indistinct. This is avoided by adding a titrating solution which overcomes the bad effects of hydrochloric acid when it is not present in large quantity.

*Solutions.*—*a.* Stannous chloride and hydrochloric acid, 1 lb. of each to 2 litres.

*b.* Hydrochloric acid 1.1 sp. gr., 1 part concentrated acid to 1 of water.

*c.* Mercuric chloride, saturated solution.

*d.* Titrating solution made by dissolving 160 grams of manganese sulphate in water and diluting to 1750 c.c., then adding 330 c.c. of phosphoric acid 1.7 sp. gr. and 320 c.c. of concentrated sulphuric acid 1.82 sp. gr.

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\* Mixer and Dubois, J. Am. Chem. Soc., Vol. XVII., p. 405.

*e.* Permanganate solution N/50 containing 6.32 grams  $K_2Mn_2O_8$  per litre.

*Standardizing.*—Dissolve two portions of about 200 mgs. each of iron wire (weighed exactly) in 20 c.c. of solution *b* in a small covered beaker. When all is dissolved expel hydrogen by boiling and then add drop by drop solution *a* until the iron is all reduced. Dilute to 100 c.c., then add at once 10 c.c. of solution *c*. This should give a white silky precipitate of  $Hg_2Cl_2$  with the excess of  $SnCl_2$ . Dilute to about 500 c.c. with cold water, add 6 to 8 c.c. of solution *d* and titrate. Read from the top of the meniscus, looking from the level of the top of the solution. After running in  $K_2Mn_2O_8$  until a drop gives to the solution a faint pink color, which remains for a minute after stirring, allow the solution to run down from the sides of the burette, then read the top of the meniscus as before.

The iron wire is usually 99.7% Fe ;

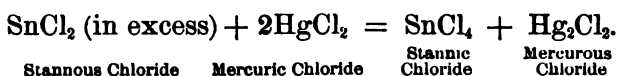
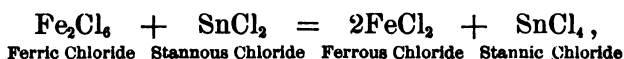
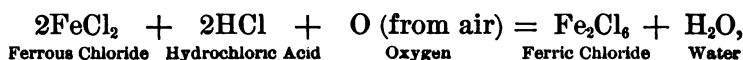
$$\frac{\text{grammes taken} \times 99.7}{\text{No. of c.c. } K_2Mn_2O_8 \text{ used}} = \text{standard.}$$

The duplicates should agree to the fifth decimal place.

*Assay.*—Grind a small quantity of the sample to an impalpable powder in an agate mortar, weigh out 50 times the standard of the  $K_2Mn_2O_8$  solution, place it in a small beaker with 3 c.c. of solution *a* and 15 c.c. of solution *b*. Cover with a watch-glass and heat till the residue is white. This does not often take more than ten minutes. When the iron is all dissolved and the solution still hot, solution *a* is added drop by drop and the operation continued as in standardizing, except that the solution *d* should be added to the water used for diluting or after the solution has



been diluted. Each c.c. of potassium permanganate ( $\text{K}_2\text{Mn}_2\text{O}_8$ ) used = 2% of iron (Fe).

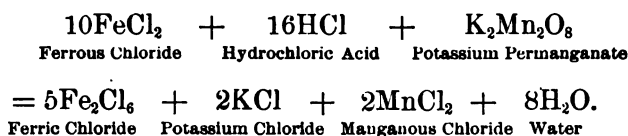


If the excess of  $\text{SnCl}_2$  is large, this reaction may take place:



In this event the analysis must be rejected.

The reaction in titration is similar to the following :



This reaction shows the proportions of  $\text{K}_2\text{Mn}_2\text{O}_8$  to Fe, but is not exactly what takes place, as hydrochloric acid (HCl), sulphuric acid ( $\text{H}_2\text{SO}_4$ ), and phosphoric acid ( $\text{H}_3\text{PO}_4$ ) are present.

The action of solution *d* is as follows : Manganous sulphate ( $\text{MnSO}_4$ ) prevents the escape of Cl; phosphoric acid ( $\text{H}_3\text{PO}_4$ ) gives a colorless solution during titration, as ferric phosphate is formed.

MANGANESE. *Symbol*—Mn.

## SOURCES.—

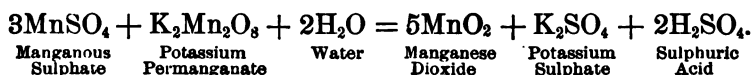
|   | Available Oxygen. | Manganese. |
|---|-------------------|------------|
| Braunite, $\text{Mn}_2\text{O}_3$ .....                     | 10%               | About 70%  |
| Pyrolusite, $\text{MnO}_2$ .....                            | 18                | 63.2       |
| Manganite, $\text{MnO}(\text{OH})$ .....                    | 9                 | 62.4       |
| Hausmannite, $\text{Mn}_3\text{O}_4$ .....                  | 6.8               | 72.0       |
| Psilomelane, $\text{MnO}_2 + \text{H}_2\text{O}$ , etc..... | Variable          |            |

Also rhodochrosite ( $\text{MnCO}_3$ ), Franklinite, wad, a mixture of oxides, and alabandite ( $\text{MnS}$ ).

Spiegeleisen and ferromanganese contain from 17 to 80% manganese.

Iron and steel commonly contain small amounts.

ASSAY.—*Volumetric Method by  $\text{K}_2\text{Mn}_2\text{O}_8$ , Volhard's.* This method is based on the following reaction which takes place in a hot dilute neutral solution containing the manganese as sulphate:



*Solution.*—A standard solution of  $\text{K}_2\text{Mn}_2\text{O}_8$  is all that is required for this method. The N/50 solution used for iron can be used for rich ores; the solution for volumetric phosphorus containing 2 grams per litre, for material low in manganese.

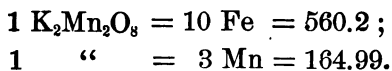
(1) *Assay of a Manganese Ore.*—Decompose 1 gram of finely ground ore with 10 c.c. conc. HCl and in some cases a little nitric acid to oxidize the iron; heat till complete decomposition is effected, then add about 25 c.c. of water and

5 to 10 c.c. of conc.  $\text{H}_2\text{SO}_4$ , and evaporate to fumes of  $\text{SO}_3$ ; cool, dilute to 100 c.c., and heat to dissolve sulphates of iron, etc. If this treatment has not completely decomposed the ore, as would be the case with Franklinite, filter off the residue and fuse with  $\text{KNaCO}_3$ , dissolve in  $\text{HCl}$ , evaporate with  $\text{H}_2\text{SO}_4$ , and add to main portion.

Nearly neutralize the sulphuric acid solution with a saturated solution of crystallized sodium carbonate, transfer to a litre flask and add a cream of oxide of zinc suspended in water until the precipitate coagulates and settles rapidly, shake after each addition of zinc oxide, allow the solution to cool, and dilute to the mark. Mix thoroughly by pouring into a large beaker and back into the flask; it makes no difference whether any is lost after mixing, as only portions are taken for titration. Filter through a dry paper into a beaker.

Take out with a pipette or flask portions of 100 or 200 c.c. each, according to the richness of the ore and the strength of the permanganate solution. Place them in casseroles and dilute with an equal volume of water. Heat to boiling, and while hot run in permanganate rapidly, stirring very vigorously with a bent rod, until a faint pink is seen against the side of the casserole after the precipitated  $\text{MnO}_2$  has settled; then stir again and see if the color is permanent, as it sometimes stirs out and requires a further addition of permanganate. Run the first portion rapidly, adding 1 c.c. of permanganate at a time near the end to get an idea of the amount required; then check by subsequent portions, adding permanganate at once to within 1 c.c. of the required amount.

Calculation :



Hence the Mn standard is  $\frac{164.99}{560.2}$  of the Fe standard, or the Fe standard multiplied by 0.294484.

(2) *Assay of Spiegel and Ferromanganese*.—Dissolve 1 gram of spiegel or 0.4 to 0.5 grams of ferromanganese in 15 c.c.  $\text{HNO}_3$  1.2 sp. gr. in a casserole, evaporate to dryness and bake to destroy combined carbon, dissolve in hydrochloric acid, add sulphuric acid, evaporate to fumes of  $\text{SO}_3$ , and proceed as described under Manganese Ore.

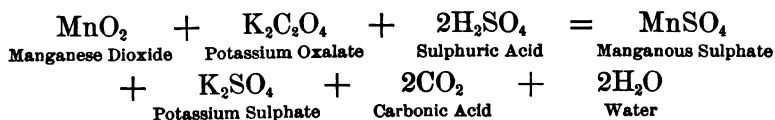
DETERMINATION OF AVAILABLE OXYGEN IN A MANGANESE ORE.

*Apparatus*.—Two flasks of about 100 c.c. capacity, provided with rubber corks fitting tight, and with two holes in each, are connected by a bent glass tube which reaches almost to the bottom of both. Through the second hole in the cork of the first flask a bent tube is placed, which can be closed at the end by a stopper made from rubber tubing and a piece of glass rod. From the second flask a glass tube conducts the escaping gas through a small tube or flask containing concentrated sulphuric acid; this is also provided with a plug to prevent the absorption of moisture while weighing. The entire apparatus must be air-tight and as light as possible. It is usually suspended by wires from the hook which holds the pans of the balance, when weighed. Any of the many forms of apparatus employed for the determination of carbonic acid, ( $\text{CO}_2$ ), by loss may be used here if of sufficient size.

*Assay*.—Weigh out accurately from 1 to 3 grams of the finely ground ore and transfer to the second (middle) flask, add about 10 grams of neutral potassium oxalate and a little water, mix by gentle agitation. Place in the first

flask about 50 c.c. of dilute sulphuric acid, and in the drying-tube or flask about 5 c.c. of concentrated sulphuric acid, so that the tube shall dip into it for half an inch ; too much acid will require a greater pressure to force the gas through, and therefore tend to make the apparatus leak. Plug the ends of the apparatus and weigh. Remove the plugs and draw some of the acid into the second flask by applying a gentle suction ; when the effervescence of carbonic acid has ceased, draw over more acid. When all the acid is over and no more carbonic acid is evolved, warm the second flask to expel any  $\text{CO}_2$  dissolved in the liquid, and draw a current of air through the flask. Allow to cool while the current passes ; when cold, plug up the ends and weigh. It is advisable to have a calcium chloride tube connected with the first flask so that the air entering the flask is dry.

*Reactions and Calculation.*



The loss in weight is due to carbonic acid ( $\text{CO}_2$ ). Each molecule of manganese dioxide gives two of  $\text{CO}_2$  ; hence  $2\text{CO}_2$  correspond to 1 of available oxygen, or 88 : 16 as the loss in weight : available oxygen, and the available oxygen divided by the weight of ore taken and multiplied by 100 gives the percentage of available oxygen.

*Remarks.*—If the ore contains carbonates, treat first with dilute sulphuric acid, boil out the  $\text{CO}_2$ , then neutralize with KOH or NaOH, and proceed as described.

NICKEL AND COBALT. *Symbols*—Ni and Co.

SOURCES.—See page 84.

ASSAY.—Nickel and cobalt are precipitated together electrolytically in the absence of the 5th and 6th groups. The conditions for precipitation are as follows: \* Nickel and cobalt present, 0.1 to 0.2 gram; ammonia 0.96 sp. gr., 100 c.c.;  $(\text{NH}_4)_2\text{SO}_4$ , 3 grams; 100 c.c. of water. Total bulk, 260 c.c. Current, 5 c.c. electrolytic gas per minute, or N.D.<sub>100</sub> 0.7 ampère (maximum).† Time required, about 4 hours at the ordinary temperature, or more rapidly at 70° C.

The method of analysis is in brief as follows: Weigh out 1 to 10 grams of ore, depending on the amount of nickel and cobalt present, decompose with nitric, hydrochloric, and sulphuric acids, evaporate to fumes, and filter. Precipitate the 5th and 6th groups by  $\text{H}_2\text{S}$ ; if only copper is present, this may be done by the current in a nitric acid solution. Separate iron by repeated ammonia precipitations or by basic acetate and ammonia precipitations. Remove zinc by precipitating with  $\text{H}_2\text{S}$  in a hot acetic acid solution, concentrate the filtrates, add ammonia and  $(\text{NH}_4)_2\text{SO}_4$ , and precipitate the nickel and cobalt together by the current; weigh, dissolve in nitric acid, neutralize with  $\text{K}_2\text{CO}_3$  or  $\text{KOH}$ , make slightly acid with acetic acid, add a strong solution of potassium nitrite ( $\text{KNO}_2$ ), and allow to stand in a warm place for 24 hours. The precipitate ( $\text{Co}_2(\text{NO})_6, 6\text{KNO}_2$ ) is yellow and crystalline; it should be washed first with a 10% solution of  $\text{KC}_2\text{H}_3\text{O}_2$ , then with alcohol; it may be treated with sulphuric acid and converted to  $3\text{K}_2\text{SO}_4 + 2\text{CoSO}_4$ , and weighed, or dissolved by  $\text{HNO}_3$ , and the cobalt precipitated by the current under the same conditions as before.

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\* Bergmann and Fresenius, Z. f. A. Ch., 19, 814.

† 0.7 ampère for every 100 sq. cm. of cathode area.

SILICON. *Symbol*—Si.

SOURCES.—As  $\text{SiO}_2$  in quartz and silicates and in alloys as silicon.

ASSAY.—Silicon is determined as silica by fusion, dehydration, etc., and also by the removal of the  $\text{SiO}_2$  by hydrofluoric acid, as is described on page 185.

With many ores where the silica is present as quartz it can be determined with considerable accuracy by treating the ore with acids and considering the insoluble residue as all silica.

When silicates are present which gelatinize, or when the ore is not readily decomposed by acids, a rapid and fairly accurate method is to fuse 1 gram of the ore with 8 to 10 grams of potassium bisulphate ( $\text{KHSO}_4$ ) (fused), at a moderate heat: this converts most of the elements to soluble sulphates, and at the same time dehydrates the silica. The fusion is leached with water and the residue treated with hot ammonium acetate to dissolve sulphate of lead, then ignited and weighed. If barium is present the silica must be determined by loss after driving it off with hydrofluoric acid ( $\text{HF}$ ), and sulphuric acid.

REMARKS.—The following properties\* of silica should be considered before a scheme for its determination in any particular case is adopted :

On adding an excess of mineral acid to a solution containing a silicate free silicic acid containing indefinite amounts of water is formed, partially or entirely soluble. On evaporation water is removed and insoluble  $\text{SiO}_2$  is separated, which may be filtered off after digestion with

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\* E. Waller, Properties of Precipitates, S. of M. Quarterly, Vol. XII.

dilute acid ; HCl is the acid most frequently used. When nitric acid is used, regard must be had for the fact that certain nitrates, ferric, etc., are decomposed at the temperature of dehydration, 110 to 130° C., and are not readily reformed by dilute nitric acid. The heat is usually maintained until there is no perceptible odor of acid. The proper temperature for dehydration is 120° C. except when sulphuric acid is used. In this case the evaporation is continued until copious fumes of  $\text{SO}_3$  are evolved.

*Solubility.*—Silica is dissolved by boiling or fusing with fixed alkalis, either carbonated or caustic.

Insoluble in water and acids except hydrofluoric.

*Contaminants.*—Lead sulphate ( $\text{PbSO}_4$ ); barium sulphate ( $\text{BaSO}_4$ ). In some cases calcium sulphate ( $\text{CaSO}_4$ ), and ferric sulphate ( $\text{Fe}_2(\text{SO}_4)_3$ ); oxide of tin ( $\text{SnO}_2$ ); oxide of antimony ( $\text{Sb}_2\text{O}_3$ ); oxide of titanium ( $\text{TiO}_2$ ); ferric oxide ( $\text{Fe}_2\text{O}_3$ ); alumina ( $\text{Al}_2\text{O}_3$ ); and basic salts.  $\text{TiO}_2$  tends to hold phosphorus pentoxide ( $\text{P}_2\text{O}_5$ ), alumina ( $\text{Al}_2\text{O}_3$ ), and ferric oxide ( $\text{Fe}_2\text{O}_3$ ), in the precipitate. To avoid error in such a case drive off the  $\text{SiO}_2$  as silicon fluoride ( $\text{SiF}_4$ ), by HF and  $\text{H}_2\text{SO}_4$  (to hold back  $\text{TiO}_2$ ).

*Ignition.*—The precipitate is very light and fine, and readily carried off by the flame. After ignition the precipitate will absorb moisture. Ignite at a high heat, and when nearly cold place in a desiccator until weighed.

### SULPHUR. *Symbol*—S.

*SOURCES.*—Native sulphur, pyrites, and numerous other sulphides.

*ASSAY.*—See methods in use for the determination of sulphur in coals and iron ores, pages 137 and 185.



For the determination of sulphur in sulphurets high in sulphur, such as pyrites, the following method may be used.

*Gladding's Method.*\*—Weigh out 1 gram of the dry and finely ground sample, place in a beaker, cover with a watch-glass, introduce 10 c.c. of bromine solution (prepared by dissolving 75 grams KBr in water, adding 50 c.c. of bromine, stirring, and diluting to 500 c.c.), mix by rotating the beaker, and allow to stand for ten minutes in the cold; add 10 c.c. nitric acid, mix again, and allow to remain ten minutes longer in the cold.

Next place the beaker on a water-bath containing cold water; heat slowly to boiling, and when the solution becomes quiet remove the glass-cover after rinsing, and evaporate to dryness; add 10 c.c. of hydrochloric acid, cover, and when violent action ceases remove cover after rinsing, and evaporate to dryness again; add 1 c.c. conc. hydrochloric acid and 50 c.c. hot water, digest till solution is complete, filter, and wash with hot water. The filtrate, about 100 c.c., is now saturated with a slight excess of ammonia and allowed to stand for ten minutes. The precipitated ferric hydroxide is filtered off and washed six times with boiling water. The filtrate is acidulated with hydrochloric acid in slight excess, heated to boiling, and 50 c.c. of barium chloride solution added, one drop per second, to the boiling liquid. The solution is allowed to stand overnight, filtered, the precipitate washed and ignited as usual.

The precipitate of ferric hydroxide is also dissolved in hot dilute hydrochloric acid, heated to boiling and 10 c.c. of barium chloride solution added. It is allowed to stand

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\* T. S. Gladding, J. Am. Chem. Soc., 1894, p. 408.

overnight and the barium sulphate thus obtained added to the main precipitate.

### ARSENIC. *Symbol*—As.

SOURCES.—The following minerals contain large quantities of arsenic:

|  |                     |
|--|---------------------|
| Realgar ( $\text{As}_2\text{S}_2$ ).....                   | 70.1 % As when pure |
| Orpiment ( $\text{As}_2\text{S}_3$ ).....                  | 61.0 “ “ “ “        |
| Arsenopyrite ( $\text{FeS}_2, \text{FeAs}_2$ ) (mispickel) | 46.0 “ “ “ “        |
| Niccolite ( $\text{NiAs}$ ).....                           | Variable            |
| Enargite ( $\text{Cu}_3\text{AsS}_4$ ).....                | 19.0 % As “ “       |
| Cobaltite ( $\text{CoAsS}$ ).....                          | 45.2 “ “ “ “        |
| Smaltite ( $(\text{CoNi})\text{As}_2$ ).....               | Variable            |
| Proustite ( $\text{Ag}_3\text{AsS}_3$ ).....               | 15.2 % As “ “       |

Arsenic is also found to a limited extent in many other minerals, artificial arsenides or speiss, and in mattes.

ASSAY.—The following method, devised by Dr. Pearce, has been found both rapid and accurate. It is a method of indirect titration, based upon the following facts: Nitrate of silver completely precipitates an alkaline arsenate in a neutral solution, as  $\text{Ag}_3\text{AsO}_4$ , soluble in nitric acid. Silver in a nitric acid solution is completely precipitated by  $\text{NH}_4\text{CNS}$ , as  $\text{AgCNS}$ . Hence for each atom of silver found there must have been  $\frac{1}{3}$  atom of arsenic in the ore taken, or for 107.92 parts of silver 25.03 parts of arsenic.

*Solutions.*—A standard solution of ammonium sulphocyanide containing about 8 grams to the litre.

A 10% solution of ferric sulphate, or a saturated solution of iron ammonia alum.

An aqueous solution of silver nitrate.

*Standardizing.*—Dissolve two portions of silver of about 200 mgs. each in nitric acid 1.16 sp. gr., dilute to 100 c.c. and add 5 c.c. of the indicator (ferric salt), run in ammonium sulphocyanide until a faint permanent red tinge is obtained. Duplicates should agree very closely. Calculate the standard of the solution for silver and for arsenic.

*Assay.*—Mix 0.25 to 1 gram of the ore with five times its weight of flux, made by mixing two parts of potassium nitrate ( $\text{KNO}_3$ ), and one of sodium carbonate ( $\text{Na}_2\text{CO}_3$ ), and place in a porcelain crucible; cover with a thin layer of potassium nitrate. Heat the crucible very gently, holding the lamp in the hand and playing the flame cautiously on the crucible; too rapid heating is attended with great danger of loss from volatilization or deflagration. During the heating no odor or white fumes of arsenic should be given off. After the fusion has become quiet continue heating for several minutes, and rotate the crucible to ensure perfect fusion and oxidation. Cool and extract the fusion with hot water, filter and wash three times with hot water. The arsenic is now in solution as alkaline arsenate. Place a piece of litmus-paper in the solution and add nitric acid ( $\text{HNO}_3$ ), carefully till acid, then a few drops in excess; boil to expel carbonic acid and nitrous fumes. Allow to cool, and neutralize by adding a large excess of an emulsion of pure zinc oxide.\* If this gives a large precipitate filter and wash, then to the filtrate add a little more zinc oxide, and while at the ordinary temperature add a slight excess of an aqueous solution of silver nitrate. If the brick-red precipitate of silver arsenate does

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\* Canby, Trans. A. I. M. E., Vol. XVII., p. 77.

not appear at once the precipitation may sometimes be hastened by adding a few drops of nitric acid, but not enough to dissolve all the zinc oxide. Filter and wash with cold water until washings are free from silver. The presence of a considerable excess of zinc oxide aids the filtration and washing.

Dissolve the precipitate through the filter with dilute nitric acid into a clean beaker, add a few c.c. of a saturated solution of ferric alum, and titrate with the standard solution of ammonium sulphocyanide ( $\text{NH}_4\text{CNS}$ ).

REMARKS.—In ores where there is danger of volatilization of arsenic before the flux melts it is well to melt a gram or two of potassium nitrate in the crucible before adding the rest of the charge.

Some ores rich in sulphur and arsenic are best treated first by heating several times with a little strong nitric acid, then evaporating to dryness with an excess of caustic alkali, very carefully to prevent spattering. The residue is mixed with half the flux and the remainder is placed on top as a cover. After fusion, the melt is treated as already described.

### PHOSPHORUS. *Symbol*—P.

SOURCES.—Apatite,  $\text{Ca}_5(\text{ClF})(\text{PO}_4)_3$ , bones, and other phosphates.

ASSAY.—For the determination of phosphorus in phosphate rocks, fertilizers, etc., the reader is referred to the bulletins of the Association of Agricultural Chemists.

For the gravimetric determination of phosphorus in iron ores see page 185.

The following method for the determination of phos-

phorus in iron and steel is both rapid and extremely accurate.

*Determination of Phosphorus by Titration of the Yellow Precipitate after Reduction, by  $K_2Mn_2O_8$ .*\*

*Reagents.*—Nitric acid sp. gr. 1.18.

Oxidizing permanganate solution, 12.5 grams per litre.

Ferrous sulphate, crystallized, free from phosphorus (Baker and Adamson).

Ammonia concentrated sp. gr. 0.9, dilute sp. gr. 0.96.

Molybdate Solution.—Dissolve 100 grams  $MoO_3$  in 400 c.c. of ammonia 0.96, filter, and pour the solution with constant stirring into 1000 c.c. nitric acid 1.2 sp. gr. Warm to  $45^\circ$  and add 1 c.c. of a 10% solution of crystallized sodium phosphate, stir vigorously, and allow to stand in a warm place overnight before using. This is to precipitate anything which might contaminate the yellow precipitate, and to leave the solution saturated with the precipitate it is afterwards to form.

Acid ammonium sulphate, 27.5 c.c., ammonia sp. gr. 0.96, and 24 c.c. concentrated  $H_2SO_4$  per litre.

Permanganate for titration, 2 grams per litre, heated for 1 hour, then filtered through asbestos, standardized against both ferrous ammonium sulphate and ammonium oxalate.

For ordinary work on pig irons, etc., the permanganate used for the titration of iron will answer every purpose.

*Jones Reductor.*—A piece of heavy glass tubing 35 cm. long and having an internal diameter of 2 cm. is drawn out so one end will pass through the cork of a filtering-flask; in the bottom is placed a perforated porcelain or platinum

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\* Noyes and Royse, J. Am. Chem. Soc., Vol. XVII., p. 129.

plate covered with asbestos. This filter should offer sufficient resistance to make a reduction occupy from 2 to 3 minutes. The tube is nearly filled with granulated zinc, about 40 mesh, which is washed with dilute sulphuric acid 1 to 20.

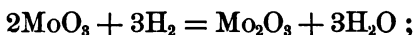
Blank determinations are made as follows : To 180 c.c. of water add 12 c.c. of ammonia 0.96 and 10 c.c. of concentrated  $\text{H}_2\text{SO}_4$ . Connect the flask with an exhaust and pass the solution through the reductor ; follow it with 200 c.c. of water, taking care that no air passes through. Titrate with permanganate in the reductor-flask and subtract the amount used from the subsequent determinations. This is done to allow for the traces of iron always present in commercial zinc. The blank should not require more than 0.3 c.c. of  $\text{K}_2\text{Mn}_2\text{O}_8$ .

*Determination in Pig Iron or Steel.*

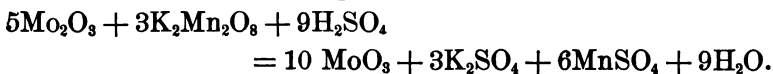
Weigh out 2 grams of steel or from 1 to 2 grams of pig iron, place in a 500-c.c. flask ; add 50 c.c.  $\text{HNO}_3$  1.18 sp. gr., and warm gently till the solution is complete ; then boil for one minute, and add 10 c.c. of the permanganate solution ; boil till the pink color disappears ; add ferrous sulphate in powder till the solution clears ; cool to about  $50^\circ$  C. and add 8 c.c. ammonia sp. gr. 0.9 ; insert a rubber stopper and shake till the precipitate of ferric hydrate and phosphate dissolves ; cool or warm the solution until it is as many degrees above or below  $60^\circ$  as the molybdate solution is below or above  $27^\circ$  C. (when mixed the temperature will be about  $45^\circ$ ). Add 60 c.c. of molybdate solution, insert the stopper, wrap up in a towel to prevent cooling, and shake vigorously for five minutes ; allow to settle for five minutes, and filter through a 9-cm. paper. Wash from the top with acid ammonium sulphate

solution until 5 c.c. give no brown color with  $\text{H}_2\text{S}$  water (showing complete removal of molybdate). Pour over the precipitate 12 c.c. of ammonia 0.96 diluted to 25 c.c.; allow the solution to run into the precipitation-flask. Wash with 100 c.c. of water; add 80 c.c. more water and 10 c.c. of conc.  $\text{H}_2\text{SO}_4$  (with a pig iron very high in phosphorus more sulphuric acid may be required). Pass the solution through the reductor; follow it with 200 c.c. of water, taking care that no air passes through the reductor during reduction or washing. Titrate the solution at once in the reductor-flask.

*Reactions and Calculations.*—In the yellow precipitate of ammonium phosphomolybdate the ratio is 12  $\text{MoO}_3$  to 1P, or as 1728 : 31. The action of the reductor is



the reaction with permanganate is



3 $\text{K}_2\text{Mn}_2\text{O}_8$  correspond to 10 $\text{MoO}_3$ .

3 $\text{K}_2\text{Mn}_2\text{O}_8$         “        “    30Fe.

Hence, Fe standard :  $\text{MoO}_3$  standard :: 30Fe : 10 $\text{MoO}_3$ ,  
or as 1680 : 1440, or as 7 : 6.

The P standard :  $\text{MoO}_3$  standard :: 31 : 1728. Hence, P standard is  $\frac{1}{54}$  of 1.794% of the Fe standard, or Fe standard multiplied by 0.01538.

This method can be used for phosphorus in an iron ore after the dehydration of the silica. Dissolve the precipitate of ferric hydroxide, etc., in nitric acid, add ammonia, and proceed exactly as with a steel.

SCHEME FOR THE DETERMINATION OF SILICA  
( $\text{SiO}_2$ ), SULPHUR (S), AND PHOSPHORUS  
(P), IN AN IRON ORE.

Weigh out from 3 to 4 grams of finely ground ore, boil with 40 c.c. concentrated HCl and 5 c.c. conc.  $\text{HNO}_3$  in a beaker as long as any solution takes place, dilute and filter; wash the residue once with dilute hydrochloric acid. Place the filtrate in a casserole to evaporate. Burn the paper containing the residue in a platinum crucible, add about six times its weight of  $\text{KNaCO}_3$  and a crystal of  $\text{NaNO}_3$ ; mix thoroughly, and fuse for about one hour over a Bunsen burner, and then over a blast-lamp. Allow the melt to cool and remove from the crucible in a cake if possible. Transfer to a casserole and cover with concentrated HCl, regulate the rate of solution by adding water, heat to boiling, and boil till everything has dissolved except perhaps some white hydrated silica. There should be no dark or gritty particles left on the bottom of the casserole; these show incomplete decomposition of the ore and have to be refused.

If the fusion is satisfactory add its solution to the first solution of the ore (which has been evaporating) and evaporate to dryness; when nearly dry stir so as to granulate the mass and present a large surface for dehydration. Place in an air-bath and heat at  $120^\circ \text{C}$ . until all hydrochloric acid (HCl), is driven out. Should the ore be very silicious this treatment must be repeated after treating or moistening again with HCl.

Moisten with 10 c.c. concentrated HCl and heat to dissolve the basic salts of iron, etc.; then dilute with water and heat to boiling, filter out the silica, and wash with dilute



HCl, then with hot water, until the washings show no chlorides. Ignite the silica at a high heat, cool, and weigh.

To make sure of the purity of the silica drive it off by hydrofluoric acid and a few drops of sulphuric acid to hold back any titanium present. Should there be any residue it may be neglected unless it is  $\text{BaSO}_4$ , in which case it must be fused with  $\text{NaKCO}_3$ , the fusion extracted with water, filtered, and the filtrate kept separate for the determination of sulphur.

To the main solution add at once a decided excess of ammonia; heat, and filter; wash the precipitate very thoroughly with boiling water, stirring up the mass each time.

*Filtrate.*—To the filtrate add the solution containing sulphur from the  $\text{BaSO}_4$  if present, acidify with HCl, heat to boiling, and while still boiling add gradually from 6 to 8 c.c. of boiling  $\text{BaCl}_2$  solution previously diluted with water; continue boiling for five minutes, filter, wash, and weigh the  $\text{BaSO}_4$ , as described on page 138.

*Precipitate.*—Transfer as much as possible of the precipitate to a beaker and place it under the funnel, pour through the paper hot dilute nitric acid till all ferric hydrate, etc., is dissolved, allowing it to run into the beaker containing the bulk of the precipitate, add more nitric acid if necessary and heat till all the precipitate has dissolved, nearly neutralize the nitric acid with ammonia, add 10 grams of ammonium nitrate and 50 c.c. of molybdate solution, transfer to a flask and shake for five minutes at a temperature of  $40^\circ \text{C}$ ., allow to stand for ten minutes and filter; wash with a 1% solution of nitric acid till  $\text{H}_2\text{S}$  gives no brown coloration to the washings. Dissolve the precipitate in dilute ammonia (1 to 4), add HCl to acid reaction, then ammonia till slightly alkaline, cool the solution, and if not

absolutely clear, filter, then add slowly drop by drop 10 c.c. of magnesia mixture, agitate the solution, and set aside in a cool place. When the precipitate of  $\text{MgNH}_4\text{PO}_4, 6\text{H}_2\text{O}$  has formed, add an excess of ammonia and allow to stand for three hours, then filter, wash with very dilute ammonia, dry the precipitate, separate it from the paper, burn the paper with the addition of nitric acid, add the precipitate, ignite strongly, and weigh the  $\text{Mg}_2\text{P}_2\text{O}_7$ .



## **PART IV.**

**LABORATORY TESTS ON ORES. ASSAY, BLOW-  
PIPE, AND QUALITATIVE SCHEMES.**



## MECHANICAL ASSAY FOR GOLD.

Crush 4 to 5 pounds of the sample, and pulverize in a mortar until it will pass through a 40 or 60 mesh sieve ; then wash in a "Batea," which is a conical pan, about 20 inches in diameter and  $2\frac{1}{2}$  in depth ; or in a Russia sheet-iron pan. The latter should be 18 to 20 inches at the top, 14 inches at the bottom, and sides 5 inches, sloping at an angle of  $30^{\circ}$  to  $40^{\circ}$ . The material to be washed should first be stirred up with water in the pan, and the sand and dirt removed by a slight circular jiggling motion, the operation being conducted under water, to hold which a small tub will be found convenient for laboratory purposes. The gold and heavier particles of the ore will collect in the bottom of the pan, and can sometimes be separated by drying, and then blowing off the sand, better than by continued washing. A magnet may prove of service here. If the ore is very poor, a "color" of gold only may be visible at the end of the operation.

## AMALGAMATION ASSAY.

Grind from 2 to 5 lbs. of ore to 80 mesh, sample, and assay. Place 6 or 8 A.T. in an iron mortar with a little water and a spoonful of clean mercury and grind with an iron pestle for from 2 to 3 hours, adding more water and a little more mercury from time to time.

The consistency of the mass must be such that the globules of mercury do not sink, but are broken up into

very fine particles. A little sodium amalgam dissolved in the mercury prevents flouring. The grinding is continued till the particles are impalpably fine. When the amalgamation is complete more water is added to reduce the mass to a thin pulp, and the stirring continued till the mercury settles to the bottom. The contents of the mortar are then washed down in a pan, the mercury collected, dried, distilled (see page 88), and the precious metals obtained by scorification, cupellation, etc.

In the case of a low-grade ore grind successive portions of ore with the same mercury to get sufficient gold to handle and to give a better average.

The tailings must be saved, dried, and assayed, and the apparent extraction checked by the gold and silver recovered.

REMARKS.—In place of the iron mortar given as the simplest form of apparatus an amalgamated copper pan or a small model amalgamation-pan may be conveniently used.

### CHLORIDIZATION ASSAY FOR SILVER.

To determine the percentage of chloridization weigh out two samples of the chloridized ore or "pulp," each  $\frac{1}{16}$  A.T. Scorify one with 30 gms. of lead, and cupel. Place the second sample in a filter-paper and wash with a strong solution of hyposulphite of soda in water (two pounds to the gallon) until all the chloride of silver in the "pulp" has been dissolved. This can be determined by adding a drop of a solution of sulphide of sodium in water to a test sample of the filtrate. When no black precipitate or brown color is formed, the chloride of silver is all dis-

solved, and the desired point has been reached. Wash the residue with pure water, dry, and burn the filter in a dish or scoop in the cupel muffle. Mix the ashes with 30 gms. of pure lead, scorify, and cupel. The calculation can best be shown by an example :

The "pulp" untreated gave 208 ounces, the "pulp" treated gave 14 ounces of silver per ton still remaining in the ore unchloridized. Hence to determine the percentage of silver chloridized, form the proportion  $208 : (208 - 14) :: 100 : x$ .

Where sulphate of silver is present a third sample leached with hot water should be run in addition to the two given above, the residue being assayed in the same way. By treating three samples the amount of silver present as sulphate, the amount contained as chloride, and the total silver can be determined.

In the West nearly all silver-reducing works make these assays every day, as the amount of chloridized silver is all that can be extracted by amalgamation.

REMARKS.—This method is sufficiently accurate to check the chloridization of the ore, and with proper care duplicate assays are unnecessary.

The operations of scorification and cupellation are conducted as already described under the head of Silver Ores.

The success of the process depends upon the care taken in washing.

If the ore is low grade larger amounts must be used.

As both sulphate and chloride of lead are somewhat soluble in "hypo" solution, sodium sulphide may give a black precipitate after all the silver chloride has been leached out.



## CHLORINATION ASSAY FOR GOLD.

The object of this assay is to determine the probable extraction of gold by the chlorination process ; it may for convenience be divided into the following steps :

- (1) Assay the original ore.
- (2) Roast a weighed quantity.
- (3) Extract the gold by chlorine.
- (4) Dry and assay the tailings.
- (5) Recover the gold dissolved.

(1) Assay 2 A.T. of the ore by any suitable crucible method.

(2) It may be necessary to determine the loss of gold in roasting. If this is to be done weigh out from 12 to 15 A.T. of the ground ore and roast in a pan or muffle to a "dead roast." Weigh the roasted ore and assay it by crucible method ; calculate the results to ounces per ton of the raw ore and note loss, if any, in roasting.

(3) Weigh out an amount of the roasted ore corresponding to 10 A.T. of the raw ore, mix this in a mortar with 10 grams of dry bleaching-powder, then transfer to a strong glass bottle of sufficient size, add warm water, and agitate the mixture until it is the consistency of thin mud. This should not fill the bottle more than half full. Add 10 grams of concentrated sulphuric acid previously diluted to 100 c.c. Place a rubber stopper in the bottle, provided with a hole through which passes a short piece of glass rod ; this will be blown out before the bottle breaks, and so act as a safety-valve. Tie down the cork, wrap the bottle in a towel, and agitate at intervals for from 4 to 5 hours. At the end of that time remove the cork, and if there is no smell of

chlorine add 10 grams more of bleaching-powder and of sulphuric acid, and agitate again for from 4 to 5 hours. If the excess of chlorine is quite strong at the end of the first five hours, add from 3 to 5 grams of bleaching-powder and the same weight of concentrated sulphuric acid instead of ten grams of each.

At the end of the second period remove the stopper again and test for free chlorine by smell or by white fumes with ammonia. To ensure the maximum extraction there must be free chlorine at the end of the operation.

If the chlorine is in excess, add more water and pour the pulp on a large filter, wash with hot water until free from chlorides.

(4) Dry the residue in an air-bath and weigh, as there will be a loss in weight which must be considered. Assay from 2 to 4 A.T. in the usual way for gold.

(5) Heat the filtrate containing gold chloride, etc., until all free chlorine is expelled, precipitate the gold by warming with ferrous sulphate or iron filings, filter out the precipitated gold, scorify, cupel, and weigh as usual.

REMARKS.—In the method given the 20 grams of bleaching-powder should give an excess of chlorine with any ordinary ore. If the assay has shown a satisfactory extraction, it must be followed by a series of tests using different quantities of bleaching-powder to determine the most economical amount. Tests should also be made to determine the proper time for leaching. In some cases comparative tests should be made on the roasted and raw ores to see whether or not the increased extraction will pay for roasting.

## BROMINE EXTRACTION TEST.

This test should indicate the percentage and amount of gold extracted by bromine solution, the amount of bromine required to combine with a ton of ore, and the rapidity of extraction. The following mode of procedure will be found to combine rapidity with a fair degree of accuracy :

Select two similar glass flasks, place in each 4 A.T. of the prepared ore and 100 c.c. of water saturated with bromine, and stopper both. Allow them to stand, with frequent agitation, one for 15 minutes and the other for 2 hours. If no test of the rapidity of the action of bromine on the ore is required, only the two-hour test need be made. If the bromine should be nearly all absorbed by the ore, indicating an enormous consumption, add sufficient pure bromine to leave a considerable excess at the end of the operation. When the digestion is finished, decant off the liquid as closely as possible through a filter into a dry beaker, and from the filtrate remove with a pipette two portions of 25 c.c. each, washing the pipette back into the flask with clean water. Now wash the ore with the minimum quantity of boiling water to remove lead bromide, and boil the filtrate and washings till all the free bromine is expelled. This filtrate represents 2 A.T. of ore. In it the bromine combined as soluble bromides is determined by means of silver nitrate either by titration with a standard solution or by weighing either the precipitated  $\text{AgBr}$  or the silver button obtained by scorifying the precipitate with test-lead and cupelling. The number of milligrams of bromine so found divided by 14.58, the number of Troy oz. in 1 lb. av. will give the number of pounds of bromine con-

sumed by two tons of ore. If chlorides are present in the ore, they should be determined and allowed for.

A comparison of the results obtained from the 15-minute and 2-hour tests will furnish a basis for judging the rapidity of the action of bromine on the ore.

One of the portions of 25 c.c. removed from the filtrate may with advantage be tested qualitatively or quantitatively for base metals, which would be precipitated as sulphides with the gold.

The percentage of extraction of the gold is determined by assaying the ore before and after treatment.

#### POTASSIUM CYANIDE EXTRACTION TEST.

The cyanide process is of great and growing importance for many low-grade ores and tailings, and laboratory tests to determine the action of cyanide on such material are often necessary. When the metallic gold is present in particles of considerable size, it should be preceded by amalgamation.

The points to be determined are:

- (1) Maximum extraction.
- (2) Strength of solution or solutions to be used.
- (3) Consumption of potassium cyanide.
- (4) Actual yield of gold and silver.

Grind several pounds of the ore to 30 or 40 mesh. Assay 2 A.T. of the ore in duplicate for gold and silver.

(1) Weigh out three portions of ore of 4 A.T. each. Place one in a bottle, add 125 c.c. of  $\frac{1}{2}\%$  cyanide solution, revolve the bottle or agitate occasionally for six hours. If the bottle is not filled more than half full, it may be corked, as there will be sufficient air present to furnish oxygen for the reaction.

Put the second portion in a beaker and leach with

water, test the washings with litmus to see whether they are acid or not, transfer ore to a bottle and treat the same as the first portion.

If the washings from the second portion were acid, to the third portion add water and agitate, then add lime to faint alkaline reaction and leach, wash and then treat with KCN as described. At the end of six hours filter and wash the pulp from each, then dry and assay the tailings. The method giving the most economical extraction is to be followed in the next tests.

(2) Wash or neutralize the ore if found advisable, and treat three portions with solutions containing 0.3, 0.2, and 0.15% KCN. Then try a very weak solution (0.1%), followed by the solution giving best extraction.

(3) Save the filtrate and washings from the two best tests, make them up to an even bulk and divide in half. Titrate half of each with a standard solution of nitrate of silver until a permanent white cloud is formed. 1  $\text{AgNO}_3$  corresponds to 2 KCN.

The reaction is as follows:  $\text{AgNO}_3 + \text{KCN} = \text{AgCN} + \text{KNO}_3$ ,  $\text{AgCN} + \text{KCN} = \text{KAg}(\text{CN})_2$ . When the  $\text{AgNO}_3$  is in excess, we have  $\text{KAg}(\text{CN})_2 + \text{AgNO}_3 = 2\text{Ag}(\text{CN})_2 + \text{KNO}_3$ .

It is best at the same time to titrate 50 c.c. of the original cyanide solution to determine the amount of cyanide actually present.

In these titrations a few c.c. of a solution of potassium iodide are sometimes added to give a more distinct end reaction. After all the cyanide has been converted into double cyanide of potassium and silver the next addition of silver nitrate gives a precipitation of yellow silver iodide ( $\text{AgI}$ ).

(4) Agitate the remaining halves of filtrates with zinc

shavings until the gold and silver are precipitated, decant, wash the gold, silver, and zinc, dissolve the excess of zinc in dilute hydrochloric acid, wash, dry, add lead, cupel, weigh, and part the gold and silver.

For further information on this subject see Rose, Metallurgy of Gold; Eissler on the Cyanide Process; Wilson, Cyanide Processes; Lodge, Cyanide Processes as Applied to the Concentrates of a Nova Scotia Gold Ore, Trans. Am. Inst. Mining Engrs., Oct., 1895.

#### RUSSELL PROCESS EXTRACTION TEST.\*

This process is extensively used for silver ores. It consists in leaching the ore, ground to from 20 to 40 mesh and usually roasted with salt, with solutions of sodium thiosulphate, "hypo," and sulphate of copper; then precipitating the precious metals with sulphide of sodium.

Two sets of tests should be made, one on the raw ore, the other on the ore after roasting alone or with from 5 to 15% of salt.

Assay the ore,—this is usually done by scorification,—then roast a portion with salt and weigh. Make up a solution containing 200 grams of crystallized copper sulphate per litre (this is called "blue" for the sake of brevity).

Weigh out portions of the ore of  $\frac{1}{2}$  A.T. each and place in beakers of 300 c.c. capacity, and treat as follows:

*a. Leaching Tests for Raw Ores and for Tailings.*—

(1) Add 250 c.c. cold water and 20 gms. hypo; let stand 12 to 16 hours cold; decant; add 25 c.c. blue; dilute to 300

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\* The Russell Process, Daggett, Trans. A. I. M. E., Feb., 1888.

c.c. with cold water ; let stand 12 to 16 hours cold ; add 20 gms. hypo ; let stand 12 to 16 hours cold ; filter and wash.

(2) Add 250 c.c. cold water and 20 gms. hypo ; let stand 12 to 16 hours cold ; add 20 gms. hypo and 25 c.c. blue ; let stand 12 to 16 hours cold ; filter and wash.

(3) Add 100 c.c. cold water and 25 c.c. blue ; let stand 1 hour cold ; add 200 c.c. cold water and 20 gms. hypo ; heat to about  $55^{\circ}$  C. =  $130^{\circ}$  F. ; filter and wash.

(4) Add 100 c.c. cold water and 25 c.c. blue ; let stand 1 hour cold ; add 200 c.c. cold water and 20 gms. hypo ; heat to about  $55^{\circ}$  C. ; filter and wash.

(5) Add 250 c.c. cold water and 25 c.c. blue ; let stand 1 hour cold ; add 20 gms. hypo ; heat on sand bath to about  $55^{\circ}$  C. ; decant ; add 200 c.c. cold water and 25 c.c. blue ; let stand 1 hour cold ; add 20 gms. hypo, heat, filter, and wash.

(6) Add 200 c.c. cold water and 90 c.c. blue ; let stand one half hour cold ; add 90 gms. hypo ; heat to about  $65^{\circ}$  C. ; filter and wash.

(7) Add 250 c.c. cold water and 25 c.c. blue ; heat to about  $55^{\circ}$  C. ; decant ; add 20 gms. hypo and 25 c.c. blue ; dilute to 300 c.c. with cold water ; heat to about  $55^{\circ}$  C. ; filter and wash.

(8) Add 200 c.c. hot water and 90 c.c. blue ; let stand one half hour ; add 90 gms. hypo ; heat to about  $65^{\circ}$  C. ; filter and wash.

*b. Leaching Tests on Roasted or Chloridized Ores, Mattes, or Tailings.*—(1) No washing ; add 50 c.c. cold water and 25 c.c. blue ; let stand 2 hours cold ; dilute to 300 c.c. with cold water ; add 20 gms. hypo ; let stand 2 hours cold ; filter and wash.

(2) No washing ; add 250 c.c. cold water and 25 c.c. blue ; let stand 12 to 18 hours cold ; add 20 gms. hypo ; let stand 12 to 18 hours cold ; filter and wash.

(3) No washing ; add 250 c.c. cold water and 25 c.c. blue ; let stand 2 hours cold ; add 20 gms. hypo ; let stand 2 hours cold ; filter and wash.

(4) Wash with cold water ; add 25 c.c. blue ; dilute to 300 c.c. with cold water ; let stand 12 to 18 hours cold ; add 20 gms. hypo ; let stand 12 to 18 hours cold ; filter and wash on filter with hot hypo solution.

(5) No washing ; add 250 c.c. cold water and 25 c.c. blue ; let stand 1 hour cold ; add 20 gms. hypo ; heat to about 55° C. ; decant ; add 250 c.c. cold water and 25 c.c. blue ; let stand 1 hour cold ; add 20 gms. hypo ; heat to about 55° C. ; filter and wash.

(6) Wash with hot water ; add 200 c.c. hot water and 90 c.c. blue ; let stand one-half hour ; add 90 gms. hypo ; heat to about 65° C. ; filter and wash.

(7) Wash with hot water ; add 90 c.c. blue ; let stand one half hour ; add 90 gms. hypo ; dilute to 300 c.c. ; heat to about 65° C. ; filter and wash.

(8) Wash with hot water ; add 20 gms. hypo ; let stand (with residue of wash-water, about 30 to 40 c.c.) 1 hour ; add 25 c.c. blue ; dilute to 300 c.c. with cold water ; heat to about 55° C. ; filter and wash.

REMARKS.—These tests have been found necessary to thoroughly test the utility of the Russell process for an ore. The yield in actual practice often varies from that obtained in a laboratory test. The solutions used in practice are much weaker. Sometimes the “hypo” and “blue” are



mixed before placing on the ore, to form the so-called "extra" solution.

In all cases the tailings are dried, assayed, and the percentage of extraction of both gold and silver calculated. The percentage of salt and time of cooling will vary with the ore and have a decided effect upon the chloridization and subsequent extraction.

For further information see Lixiviation of Silver Ores with Hyposulphite Solutions, with special reference to the Russell process, Carl A. Stetefeldt, Oakland, Cal.

## OUTLINE SCHEME FOR THE ASSAY OF ORES.

The ore may contain gold, silver, lead, antimony, bismuth, tin, mercury, zinc, platinum, copper, iron, nickel, cobalt, sulphur, arsenic, tellurium, selenium, etc. Required to determine gold, silver, and useful metals.

*a.* Inspect with magnifying-glass. Test with blowpipe. Sample, pulverize, and sift. If scales remain on sieve, test with magnet, as they may come from mortar; if not magnetic, weigh residue and siftings, and assay separately. Calculate value as directed, page 104.

*b.* Treat scales by scorification for gold and silver. For other metals treat as an alloy by wet methods.

*c.* Charge: ore 10 gms., cyanide of potassium 40 gms., in No. 5 crucible, and fuse in a quick fire; cool, break, and weigh. The button may contain lead, antimony, bismuth, copper, tin, iron, etc. Test by blowpipe. If lead, see page 64. If tin, antimony, or bismuth, duplicate by cyanide fusion. If iron, run charges for unknown ores, page 76. If tin is present, it will not be necessary, as a

rule, to look for other metals. The button from the cyanide fusion should, however, be treated as an alloy.

d. Run preliminary assay (see page 92); weigh lead button to determine reducing power (R.P.); cupel the lead button to determine approximate richness of ore. Notice the color of cupel, to decide as to presence of copper. The reducing power may be :

No. 1. No reducing power. The ore is therefore free from sulphurets, arsenic, antimony, etc.

(a) If the preliminary assay has shown it to be rich in silver, run by scorification assay, using  $\frac{1}{2}$  A.T. ore, 40 grams test-lead, and borax glass to suit the gangue.

(b) If poor in silver, run by crucible assay. Ore, 2 A.T.; litharge, 2 A.T.; soda, 2 A.T.; argol from 2 to 4 grams; borax glass, 10 grams; silica, from 0 to 2 A.T., depending on the gangue; salt cover.

No. 2. Reducing power low, lead reduced by 5 grams of ore from 0 to 8 grams.

(a) Rich in silver, scorification assay. Ore,  $\frac{1}{2}$  A.T.; test-lead, 50 grams; borax glass as required.

(b) Poor in silver, crucible assay. Ore, 1 or 2 A.T.; PbO, 2 A.T.; soda, 2 A.T.; borax glass, 10 grams; silica, from 0 to 2 A.T.; salt cover, and nitre or argol as calculated from result of preliminary assay. Or run several scorifications, combine the lead buttons, and rescorify before cupellation.

No. 3. Reducing power high, lead reduced by 5 grams of ore more than 8 grams.

The ore probably contains large quantities of sulphur, arsenic, antimony, etc.

(a) Rich in silver, scorification assay. Ore,  $\frac{1}{2}$  or  $\frac{1}{10}$  A.T.;

test-lead, 60 grams ; borax glass as required. It may be advantageous to roast the ore first in the scorifier.

(b) Poor in silver, crucible assay. Roast 2 A.T., then add litharge, 3 A.T.; soda, 2 A.T.; silica, from 1 to  $2\frac{1}{2}$  A.T.; borax glass, 10 grams ; argol, from 2 to 4 grams ; salt cover. Or ore, 1 A.T.; litharge, 2 A.T.; soda, 1 A.T.; nitre calculated to give a button of 18 grams ; silica, 1 A.T.; salt cover.

See special charges given on page 106.

e. If button freezes in process of cupellation, the furnace being hot, and the cupel unsaturated, copper, cobalt, nickel, tin, or platinum may be present ; cupel dark brown or red after cupellation, probably copper.

1. Determine cobalt, nickel, and copper by arsenide method, page 84.

2. For platinum treat button as an alloy, page 131.

3. Tin : run special assay, section c.

f. If the preliminary blowpipe test indicates the presence of mercury, zinc, or tellurium, etc., the ore should be run by scorification method for gold and silver. For ores containing mercury the heat should be *very low* at first, and increased *gradually*. For tellurides, see page 107.

The zinc and mercury may be determined by the methods given on pages 152 and 87 ; or, if the ores are impure, by gravimetric methods.

REMARKS.—The above scheme is designed as a guide for beginners. The experienced mineralogist and assayer will generally be able to determine the character of the ore by inspection, and select his method.

## BLOWPIPE APPARATUS AND REAGENTS.

The following list includes the apparatus and reagents essential for ordinary qualitative blowpipe work :

Set of three porcelain dishes.

Diamond steel mortar.

3 pairs of forceps, 1 platinum-pointed, 1 ordinary steel,  
1 heavy steel.

Steel chisel and magnet.

2 charcoal-borers, 1 four-cornered with spatula, 1 club-shaped.

Pair lamp scissors.

1 wire-holder with five platinum wires.

Plattner's blowpipe lamp with swivel, nickel plated.

Charcoal-saw.

Matrass-holder.

Nickel-plated Plattner's blowpipe with platinum tips.

Steel hammer.

Set of moulds and stamps for cupels.

Pair steel nippers.

Double pocket-lens.

Camel's-hair brush.

Dropping-pipette.

Ivory spoon.

1 dozen matrasses.

Alcohol lamp with nickel-plated top.

Chamois-skin.

6 glass tubes.

1 dozen pieces square-cut charcoal.

6 pieces plaster for coats.

Porcelain plate for streaks.

Metal tray for coal.

Metal tray for ashes.

6 small watch-glasses.

Mixing-scoop.

18 reagent-bottles in a frame, filled with the following reagents: Test-lead, tin granulated, phosphorus salt, borax powdered, borax glass, boracic acid fused, Plattner's flux, bismuth flux, boracic acid, sodium carbonate, salt, sodium nitrate, charcoal, bone-ash sieved, bone-ash washed, copper oxide, potassium bisulphate.

Also, small glass-stoppered bottles containing cobalt nitrate, hydrochloric acid, and nitric acid.

Cost with a mahogany case about \$40.00.

#### REMARKS.

To clean a dirty platinum point, hold it in the flame of the alcohol lamp with the platinum-pointed forceps.

To clean platinum wires, heat, and plunge into muriatic acid while hot.

To break small pieces of mineral, wrap in paper, tin-foil, or cloth before hammering.

To trim the wick of blowpipe lamp, cut even with the lamp and raise with one point of the steel forceps; never pull the wick up with the forceps.

To light the usual blowpipe lamp where a heavy oil is used, direct the flame of an alcohol lamp against the wick until it ignites.

## BLOWPIPE TESTS.\*

In the following test the details of ordinary manipulations, such as obtaining beads, flames, coatings, and sublimates, are omitted and the results alone stated; unusual manipulations are described. The bead tests are supposed to be obtained with oxides; the other tests are in general true of all compounds not expressly excluded. The course to be followed in the case of interfering elements is briefly stated.

## Aluminum, Al.

*With Soda.*—Swells and forms an infusible compound.

*With Borax or S. Ph.*—Clear or cloudy, never opaque.

*With Cobalt Solution.*†—Fine blue when cold.

Ammonium,  $\text{NH}_4$ .

*In Closed Tube.*—Evolution of gas with the characteristic odor. Soda or lime assists the reaction. The gas ( $\text{NH}_3$ ) turns red litmus-paper blue and forms white clouds with HCl vapor.

## Antimony, Sb.

*On Coal, R.F.*‡—Volatile white coat, bluish in thin layers, continues to form after cessation of blast and appears to come directly off the mass.

*With Bismuth Flux:*

*On Plaster.*—Peach-red coat, somewhat mottled.

*On Coal.*—Faint yellow or red coat.

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\* Mineralogy, Crystallography and Blowpipe Analysis by Moses and Parsons.

† Certain phosphates, borates, and fusible silicates become blue in absence of alumina.

‡ This coat may be further tested by S. Ph. or flame.

*In Open Tube.*—Dense, white, non-volatile, amorphous sublimate. The sulphide too rapidly heated will yield spots of red.

*In Closed Tube.*—The oxide will yield a white fusible sublimate of needle crystals; the sulphide, a black sublimate, red when cold.

*Flame.*—Pale yellow-green.

*With S. Ph.*—Dissolved by O. F. and treated on coal with tin in R. F. becomes gray to black.

#### INTERFERING ELEMENTS.

*Arsenic.*—Remove by gentle O. F. on coal.

*Arsenic with Sulphur.*—Remove by gently heating in closed tube.

*Copper.*—The S. Ph. bead with tin in R. F. may be momentarily red, but will blacken.

*Lead or Bismuth.*—Retard formation of their coats by intermittent blast or by boracic acid. Confirm coat by flame, not by S. Ph.

#### Arsenic, As.

*On Smoked Plaster.*—White coat of octahedral crystals.

*On Coal.*—Very volatile white coat and strong garlic odor. The oxide and sulphide should be mixed with soda.

*With Bismuth Flux:*

*On Plaster.*—Reddish orange coat, made yellow by  $(\text{NH}_4)_2\text{S}$ .

*On Coal*—Faint yellow coat.

*In Open Tube.*—White sublimate of octahedral crystals. Too high heat may form deposit of red or yellow sulphide.

*In Closed Tube.*—May obtain white oxide, yellow or red sulphide, or black mirror of metal.

*Flame.*—Pale azure blue.

## INTERFERING ELEMENTS.

*Antimony*.—Heat in closed tube with soda and charcoal, treat resulting mirror in O. F. for odor.

*Cobalt or Nickel*.—Fuse in O. F. with lead and recognize by odor.

*Sulphur*.—(a) Red to yellow sublimate of sulphide of arsenic in closed tube.

(b) Odor when fused with soda on coal.

**Barium, Ba.**

*On Coal with Soda*.—Fuses and sinks into the coal.

*Flame*.—Yellowish green, improved by moistening with HCl.

*With Borax or S. Ph.*—Clear and colorless, can be flamed opaque white.

**Bismuth, Bi.**

*On Coal*.—In either flame is reduced to brittle metal and yields a volatile coat, dark orange yellow hot, lemon-yellow cold, with yellowish-white border.

*With Bismuth Flux*: \*

*On Plaster*.—Bright scarlet coat surrounded by chocolate-brown, with sometimes a reddish border.

The brown may be made red by ammonia.†

*On Coal*.—Bright red coat with sometimes an inner fringe of yellow.

*With S. Ph.*—Dissolved by O. F. and treated on coal with tin in R. F. is colorless hot, but blackish gray and opaque cold.

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\* Sulphur 2 parts, potassium iodide 1 part, potassium bisulphate 1 part.

† May be obtained by heating S. Ph. on the assay.



## INTERFERING ELEMENTS.

*Antimony*.—Treat on coal with boracic acid, and treat the resulting slag on plaster with bismuth flux.

*Lead*.—Dissolve coat in S. Ph. as above.

**Boron, B.**

All borates intumesce and fuse to a bead.

*Flame*.—Yellowish green. May be assisted by (a) moistening with  $H_2SO_4$ ; (b) mixing to paste with water and boracic acid flux ( $4\frac{1}{2}$  pts.  $KHSO_4$ , 1 pt.  $CaF_2$ ); (c) by mixing to paste with  $H_2SO_4$  and  $NH_4F$ .

**Bromine, Br.**

*With S. Ph. Saturated with CuO*.—Treated at tip of blue flame, the bead will be surrounded by greenish-blue flames.

*In Matrass with  $KHSO_4$* .—Brown choking vapor.

## INTERFERING ELEMENTS.

*Silver*.—The bromide melts in  $KHSO_4$  and forms a blood-red globule which cools yellow and becomes green in the sunlight.

**Cadmium, Cd.**

*On Coal R. F.*—Dark brown coat, greenish yellow in thin layers. Beyond the coat, at first part of operation, the coal shows a variegated tarnish.

*On Smoked Plaster with Bismuth Flux*.—White coat made orange by  $(NH_4)_2S$ .

*With Borax or S. Ph.*—O. F. Clear yellow hot, colorless cold, can be flamed milk-white. The hot bead touched to  $Na_2S_2O_3$  becomes yellow.

R. F. Becomes slowly colorless.

## INTERFERING ELEMENTS.

*Lead, Bismuth, Zinc.*—Collect the coat, mix with charcoal-dust, and heat gently in a closed tube. Cadmium will yield either a reddish-brown ring or a metallic mirror. Before collecting coat treat it with O. F. to remove arsenic.

## Calcium, Ca.

*On Coal with Soda.*—Insoluble and not absorbed by the coal.

*Flame.*—Yellowish red, improved by moistening with HCl.

*With Borax or S. Ph.*—Clear and colorless, can be flamed opaque.

Carbonic Acid, CO<sub>2</sub>.

*With Nitric Acid.*—Heat with water and then with dilute acid. CO<sub>2</sub> will be set free with effervescence. The escaping gas will render limewater turbid.

*With Borax or S. Ph.*—After the flux has been fused to a clear bead the addition of a carbonate will cause effervescence during further fusion.

## Chlorine, Cl.

*With S. Ph. Saturated with CuO.*—Treated at tip of blue flame, the bead will be surrounded by an intense azure-blue flame.

*On Coal with CuO.*—Grind with a drop of H<sub>2</sub>SO<sub>4</sub>, spread the paste on coal, dry gently in O. F. and treat with blue flame, which will be colored greenish blue and then azure-blue.

**Chromium, Cr.**

*With Borax or S. Ph.*—O. F. Reddish hot, fine yellow-green cold.

R. F. In borax, green hot and cold. In S. Ph. red hot, green cold.

*With Soda.*—O. F. Dark yellow hot, opaque and light yellow cold.

R. F. Opaque and yellowish green cold.

## INTERFERING ELEMENTS.

*Manganese.*—The soda bead in O. F. will be bright yellowish green.

**Cobalt, Co.**

*On Coal R. F.*—The oxide becomes magnetic metal. The solution in HCl will be rose-red, but on evaporation will be blue.

*With Borax or S. Ph.*—Pure blue in either flame.

## INTERFERING ELEMENTS.

*Arsenic.*—Roast and scorify with successive additions of borax. There may be, in order given, yellow (iron), green (iron and cobalt), blue (cobalt), reddish brown (nickel), green (nickel and copper), blue (copper).

*Copper and Other Elements which Color Strongly.*—Fuse with borax and lead on coal in R. F. The borax on platinum wire in O. F. will show the cobalt, except when obscured by much iron or chromium.

*Iron, Nickel, or Chromium.*—Fuse in R. F. with a little metallic arsenic, then treat as an arsenide.

*Sulphur or Selenium.*—Roast and scorify with borax, as before described.

**Copper, Cu.**

*On Coal R. F.*—Formation of red malleable metal.

*Flame.\**—Emerald-green or azure-blue, according to compound.

The azure-blue flame may be obtained :

(a) By moistening with HCl or aqua regia, drying gently in O. F., and heating strongly in R. F.

(b) By saturating S. Ph. bead with substance, adding common salt, and treating with blue flame.

*With Borax † or S. Ph.*—O. F. Green hot, blue or greenish blue cold.

R. F. Greenish or colorless hot, opaque and brownish red cold. With tin on coal this reaction is more delicate.

**INTERFERING ELEMENTS.**

*General Method.‡*—Roast thoroughly, treat with borax on coal in strong R. F., and

*If Button Forms.*—Separate the button from the slag, remove any lead from it by O. F., and make either S. Ph. or flame test upon residual button.

*If No Visible Button Forms.*—Add test-lead to the borax fusion, continue the reduction, separate the button, and treat as in next test (Lead Alloy).

*Lead or Bismuth Alloys.*—Treat with frequently

\*Sulphur, selenium, and arsenic should be removed by roasting. Lead necessitates a gentle heat.

†By repeated slow oxidation and reduction a borax bead becomes ruby-red.

‡Oxides, sulphides, and sulphates are best reduced by a mixture of soda and borax.

changed boracic acid in strong R. F., noting the appearance of slag and residual button.

*Trace.*—A red spot in the slag.

*Over One Per Cent.*—The residual button will be bluish green when melted, will dissolve in the slag and color it red upon application of the C. F., or may be removed from the slag and be submitted to either the S. Ph. or the flame test.

### Fluorine, F.

*Etching Test.*—If fluorine is released, it will corrode glass in cloudy patches, and in presence of silica there will be a deposit on the glass. According to the refractoriness of the compound the fluorine may be released :

- (a) In closed tube by heat.
- (b) In closed tube by heat and  $\text{KHSO}_4$ .
- (c) In open tube by heat and glass of S. Ph.

*With Conc.  $\text{H}_2\text{SO}_4$  and  $\text{SiO}_2$ .*—If heated and the fumes condensed by a drop of water upon a platinum wire, a film of silicic acid will form upon the water.

### Iodine, I.

*With S. Ph. Saturated with  $\text{CuO}$ .*—Treated at the tip of the blue flame, the bead is surrounded by an intense emerald-green flame.

*In Matrass with  $\text{KHSO}_4$ .*—Violet choking vapor and brown sublimate.

*In Open Tube with Equal Parts Bismuth Oxide, Sulphur, and Soda.*—A brick-red sublimate.

*With Starch-paper.*—The vapor turns the paper dark purple.

## INTERFERING ELEMENTS.

*Silver*.—The iodide melts in  $\text{KHSO}_4$  to a dark red globule, yellow on cooling, and unchanged by sunlight.

## Iron, Fe.

*On Coal*.—R. F. Many compounds become magnetic. Soda assists the reaction.

*With Borax*.\*—O. F. Yellow to red hot, colorless to yellow cold.

R. F. Bottle-green. With tin on coal vitriol-green.

*With S. Ph.*—O. F. Yellow to red hot, greenish while cooling, colorless to yellow cold.

R. F. Red hot and cold, greenish while cooling.

*State of the Iron*.—A borax bead blue from  $\text{CuO}$  is made red by  $\text{FeO}$ , and greenish by  $\text{Fe}_2\text{O}_3$ .

## INTERFERING ELEMENTS.

*Chromium*.—Fuse with nitrate and carbonate of soda on platinum, dissolve in water, and test residue for iron.

*Cobalt*.—By dilution the blue of cobalt in borax may often be lost before the yellow of iron.

*Copper*.—May be removed from borax bead by fusion with lead on coal in R. F.

*Manganese*.—(a) May be faded from borax bead by treatment with tin on coal in R. F.

(b) May be faded from S. Ph. bead by R. F.

*Nickel*.—May be faded from borax bead by R. F.

*Tungsten or Titanium*.—The S. Ph. bead in R. F. will be reddish brown instead of blue or violet.

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\* A slight yellow color can only be attributed to iron when there is no decided color produced by either flame in highly charged beads of borax and S. Ph.

*Uranium*.—As with chromium.

*Alloys, Sulphides, Arsenides, etc.*—Roast, treat with borax on coal in R. F., then treat borax in R. F. to remove reducible metals.

### Lead, Pb.

*On Coal*.\*—In either flame is reduced to malleable metal and yields, near the assay, a dark lemon-yellow coat, sulphur-yellow cold and bluish white at border.

*With Bismuth Flux*:

*On Plaster*.—Chrome-yellow coat, blackened by  $(\text{NH}_4)_2\text{S}$ .

*On Coal*.—Volatile yellow coat, darker hot.

*Flame*.—Azure-blue.

*With Borax or S. Ph.*—O. F. Yellow hot, colorless cold, flames opaque yellow.

R. F. Borax bead becomes clear, S. Ph. bead cloudy.

### INTERFERING ELEMENTS.

*Antimony*.—Treat on coal with boracic acid, and treat the resulting slag on plaster with bismuth flux.

*Arsenic Sulphide*.—Remove by gentle O. F.

*Cadmium*.—Remove by R. F.

*Bismuth*.—Usually the bismuth-flux tests on plaster are sufficient. In addition the lead coat should color the R. F. blue.

### Lithium, Li.

*Flame*.—Crimson, best obtained by gently heating near the wick.

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\* The phosphate yields no coat without the aid of a flux.

## INTERFERING ELEMENTS.

*Sodium.*—(a) Use a gentle flame and heat near the wick. (b) Fuse on platinum wire with barium chloride in O. F. The flame will be first strong yellow, then green, and lastly crimson.

*Calcium or Strontium.*—As these elements do not color the flame in the presence of barium chloride, the above test will answer.

*Silicon.*—Make into a paste with boracic acid flux and water, and fuse in the blue flame. Just after the flux fuses the red flame will appear.

**Magnesium, Mg.**

*On Coal with Soda.*—Insoluble, and not absorbed by the coal.

*With Borax or S. Ph.*—Clear and colorless, can be flamed opaque white.

*With Cobalt Solution.\**—Strongly heated becomes a pale flesh color.

**Manganese, Mn.**

*With Borax or S. Ph.†*—O. F. Amethystine hot, red-dens on cooling. With much, is black and opaque. If a hot bead is touched to a crystal of sodium nitrate, an amethystine or rose-colored froth is formed.

R. F. Colorless or with black spots.

*With Soda.*—O. F. Bluish green and opaque when cold. Sodium nitrate assists the reaction.

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\* With silicates this reaction is of use only in the absence of coloring oxides. The phosphate, arsenate, and borate become violet-red.

† The colors are more intense with borax than with S. Ph.



## INTERFERING ELEMENTS.

*Chromium.*—The soda bead in O. F. will be bright yellowish green instead of bluish green.

*Silicon.*—Dissolve in borax, then make soda fusion.

**Mercury, Hg.**

*With Bismuth Flux:*

*On Plaster.*—Volatile yellow and scarlet coat. If too strongly heated, the coat is black and yellow.

*On Coal.*—Faint yellow coat at a distance.

*In Matrass with Dry Soda or with Litharge.\**—Mirror-like sublimate, which may be collected in globules.

**Molybdenum, Mo.**

*On Coal.*—O. F. A coat yellowish hot, white cold, crystalline near assay.

R. F. The coat is turned in part deep blue, in part dark copper-red.

*Flame.*—Yellowish green.

*With Borax.*—O. F. Yellow hot, colorless cold.

R. F. Brown to black and opaque.

*With S. Ph.*—O. F. Yellowish green hot, colorless cold.†

R. F. Emerald-green.

*Dilute ( $\frac{1}{4}$ ) HCl Solutions.*—If insoluble the substance may first be fused with S. Ph. in O. F. If then dissolved in the acid and heated with metallic tin, zinc, or copper, the solutions will be successively blue, green, and brown. If the S. Ph. bead has been treated in R. F., the solution will become brown.

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\* Gold-leaf is whitened by the slightest trace of vapor of mercury.

† Crushed between damp unglazed paper becomes red, brown, purple, or blue, according to amount present.

**Nickel, Ni.**

*On Coal.*—R. F. The oxide becomes magnetic.

*With Borax.*—O. F. Violet hot, pale reddish brown cold.

R. F. Cloudy and finally clear and colorless.

*With S. Ph.*—O. F. Red hot, yellow cold.

R. F. Red hot, yellow cold. On coal with tin becomes colorless.

**INTERFERING ELEMENTS.**

*General Method.*—Saturate two or three borax beads with roasted substance, and treat on coal with a strong R. F. If a visible button results, separate it from the borax, and treat with S. Ph. in the O. F., replacing the S. Ph. when a color is obtained.

If no visible button results, add either a small gold button or a few grains of test-lead. Continue the reduction, and :

*With Gold.*—Treat the gold alloy on coal with S. Ph. in strong O. F.

*With Lead.*—Scorify button with boracic acid to small size, complete the removal of lead by O. F. on coal, and treat residual button with S. Ph. in O. F.

*Arsenic.*—Roast thoroughly, treat with borax in R. F. as long as it shows color, treat residual button with S. Ph. in O. F.

*Alloys.*—Roast and melt with frequently changed borax in R. F., adding a little lead if infusible. When the borax is no longer colored, treat residual button with S. Ph. in O. F.

**Nitric Acid,  $\text{HNO}_3$ .**

*In Matrass with  $\text{KHSO}_4$ , or in Closed Tube with Litharge.*—Brown fumes with characteristic odor. The fumes will turn ferrous sulphate paper brown.

**Phosphorus, P.**

*Flame.*—Greenish blue, momentary. Improved by conc.  $\text{H}_2\text{SO}_4$ .

*In Closed Tube with Dry Soda and Magnesium.*—The soda and substance are mixed in equal parts and dried, and made to cover the magnesium. Upon strongly heating there will be a vivid incandescence, and the resulting mass, crushed and moistened, will yield the odor of phosphuretted hydrogen.

**Potassium, K.**

*Flame.*—Violet, except borates and phosphates.

**INTERFERING ELEMENTS.**

*Sodium.*—(a) The flame, through blue glass, will be violet or blue.

(b) A bead of borax and a little boracic acid, made brown by nickel, will become blue on addition of a potassium compound.

*Lithium.*—The flame, through green glass, will be bluish green.

**Selenium, Se.**

*On Coal R. F.*—Disagreeable horse-radish odor, brown fumes, and a volatile steel-gray coat with a red border.

*In Open Tube.*—Steel-gray sublimate with red border, sometimes white crystals.

*In Closed Tube.*—Dark red sublimate and horse-radish odor.

*Flame.*—Azure-blue.

*On Coal with Soda.*—Thoroughly fuse in R. F., place on bright silver, moisten, crush, and let stand. The silver will be blackened.

### Silicon, Si.

*On Coal with Soda.*—With its own volume of soda dissolves with effervescence to a clear bead. With more soda the bead is opaque.

*With Borax.*—Clear and colorless.

*With S. Ph.*—Insoluble. The test made upon a small fragment will usually show a translucent mass of undissolved matter of the shape of the original fragment.

When not decomposed by S. Ph., dissolve in borax nearly to saturation, add S. Ph., and reheat for a moment. The bead will become milky or opaque white.

### Silver, Ag.

*On Coal.*—Reduction to malleable white metal.

*With Borax or S. Ph.*—O. F. Opalescent.

*Cupellation.*—Fuse on coal with 1 vol. of borax glass and 1 to 2 vols. of test-lead in R. F. for about two minutes. Remove button and scorify it in R. F. with fresh borax, then place button on cupel and blow O. F. across it, using as strong blast and as little flame as are consistent with keeping button melted.

If the litharge is dark, or if the button freezes before brightening, or if it brightens but is not spherical, rescorify it on coal with borax, add more test-lead, and again cupel until there remains only a white spherical button of silver.

**Sodium, Na.**

*Flame*.—Strong reddish yellow.

**Strontium, Sr.**

*On Coal with Soda*.—Insoluble, absorbed by the coal.

*Flame*.—Intense crimson, improved by moistening with HCl.

*With Borax or S. Ph.*—Clear and colorless; can be flamed opaque.

**INTERFERING ELEMENTS.**

*Barium*.—The red flame may show upon first introduction of the sample into the flame, but it is afterward turned brownish yellow.

*Lithium*.—Fused with barium chloride, by which the lithium flame is unchanged.

**Sulphur, S.**

*On Coal with Soda and a Little Borax*.—Thoroughly fuse in the R. F., and either:

- (a) Place on bright silver, moisten, crush, and let stand. The silver will become brown to black. Or,
- (b) Heat with dilute HCl (sometimes with powdered zinc); the odor of  $H_2S$  will be observed.

*In Open Tube*.—Suffocating fumes. Some sulphates are unaffected.

*In Closed Tube*.—May have sublimate red when hot, yellow cold, or sublimate of undecomposed sulphide, or the substance may be unaffected.

*With Soda and Silica* (equal parts).—A yellow or red bead.

*To Determine Whether Sulphide or Sulphate.*—Fuse with soda on platinum-foil. The sulphide only will stain silver.

### Tellurium, Te.

*On Coal.*—Volatile white coat with red or yellow border. If the fumes are caught on porcelain, the resulting gray or brown film may be turned crimson when moistened with conc.  $H_2SO_4$  and gently heated.

*On Coal with Soda.*—Thoroughly fuse in R. F. Place on bright silver, moisten, crush, and let stand. The silver will be blackened.

*Flame.*—Green.

*In Open Tube.*—Gray sublimate fusible to clear drops.

*With  $H_2SO_4$  (conc.).*—Boiled a moment, there results a purple-violet solution, which loses its color on further heating or on dilution.

### Tin, Sn.

*On Coal.*—O. F. The oxide becomes yellow and luminous.

R. F. A slight coat, assisted by addition of sulphur or soda.

*With Cobalt Solution.*—Moisten the coal, in front of the assay, with the solution, and blow a *strong* R. F. upon the assay. The coat will be bluish green when cold.

*With  $CuO$  in Borax Bead.*—A faint blue bead is made reddish brown or ruby-red by heating a moment in R. F. with a tin compound.

### INTERFERING ELEMENTS.

*Lead or Bismuth (Alloys).*—It is fair proof of tin if such an alloy oxidizes rapidly with sprouting and cannot be kept fused.

*Zinc.*—On coal with soda, borax, and charcoal in R. F. the tin will be reduced, the zinc volatilized; the tin may then be washed from the fused mass.

### Titanium, Ti.

*With Borax.*—O. F. Colorless to yellow hot, colorless cold, opalescent or opaque white by flaming.

R. F. Yellow to brown, enamel blue by flaming.

*With S. Ph.*—O. F. As with borax.

R. F. Yellow hot, violet cold.

*HCl Solutions.*—If insoluble, the substance may first be fused with S. Ph. or with soda and reduced. If then dissolved in dilute acid and heated with metallic tin, the solution will become violet after standing. Usually there will also be a turbid violet precipitate, which becomes white.

### INTERFERING ELEMENTS.

*Iron.*—The S. Ph. bead in R. F. is yellow hot, brownish red cold.

### Tungsten, W.

*With Borax.*—O. F. Colorless to yellow hot, colorless cold, can be flamed opaque white.

R. F. Colorless to yellow hot, yellowish brown cold.

*With S. Ph.*—O. F. Clear and colorless.

R. F. Greenish hot, blue cold. On long blowing or with tin on coal becomes dark green.

*With Dilute HCl.*—If insoluble, the substance may first be fused with S. Ph. The solution heated with tin becomes dark blue; with zinc it becomes purple and then reddish brown.

## INTERFERING ELEMENTS.

*Iron*.—The S. Ph. in R. F. is yellow hot, blood-red cold.

**Uranium, U.**

*With Borax*.—O. F. Yellow hot, colorless cold, can be flamed enamel yellow.

R. F. Bottle-green, can be flamed black, but not enamelled.

*With S. Ph.*—O. F. Yellow hot, yellowish green cold.

R. F. Emerald-green.

## INTERFERING ELEMENTS.

*Iron*.—With S. Ph. in R. F. is green hot, red cold.

**Vanadium, V.**

*With Borax*.—O. F. Colorless or yellow hot, greenish yellow cold.

R. F. Brownish hot, emerald-green cold.

*With S. Ph.*—O. F. Dark yellow hot, light yellow cold.

R. F. Brown hot, emerald-green cold.

*H<sub>2</sub>SO<sub>4</sub> Solutions*.—Reduced by Zn become successively yellow, green, bluish green, blue, greenish blue, bluish violet, and lavender.

**Zinc, Zn.**

*On Coal*.—O. F. The oxide becomes yellow and luminous.

R. F. Yellow coat, white when cold, assisted by soda and a little borax.

*With Cobalt Solution*.—Moisten the coal, in front of



the assay, with the solution, and blow a strong R. F. upon the assay. The coat will be bright yellow-green when cold.

#### INTERFERING ELEMENTS.

*Antimony*.—Remove by strong O. F., or by heating with sulphur in closed tube.

*Cadmium, Lead, or Bismuth*.—The combined coats will not prevent the cobalt solution test.

*Tin*.—The coats, heated in an open tube, with charcoal-dust, by the O. F., may yield white sublimate of zinc.

#### SCHEMES FOR QUALITATIVE BLOWPIPE ANALYSIS.\*

TEST I.—Heat a portion gently with O. F. upon charcoal or a plaster tablet which has been blackened in the lamp flame.

**As.**—White very volatile crystalline coat, best on plaster.

The coat disappears before R. F., tingeing it pale blue and evolving a characteristic garlic odor.

*Confirmation As.*—The coating may be dissolved in solution of KOH, placed in a test-tube, a small piece of sodium amalgam added, and the tube covered with a piece of filter-paper moistened with a *slightly* acid solution of  $\text{AgNO}_3$ . The paper will be stained black by the  $\text{AsH}_3$  evolved.

**Sb.**—White pulverulent volatile coat, best on charcoal.

A good distinguishing feature between As and Sb is as

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\* These schemes are taken from Mineralogy, Crystallography, and Blowpipe Analysis, by A. J. Moses and C. L. Parsons, D. Van Nostrand, 1895.

follows: They both usually continue to give off fumes after removal of the flame, but while still hot the  $\text{As}_2\text{O}_3$  fumes are not visible within one half inch of assay, while  $\text{Sb}_2\text{O}_3$  fumes appear to come immediately off of the mass.

*Confirmation Sb.*—The coating disappears before R. F., tingeing it a pale yellow-green, or, if scraped together, dissolved in S. Ph., and *just* fused on charcoal in contact with tin, it will form a gray or black opaque bead.

If the coating be scraped off and dissolved in tartaric acid and HCl, and the solution placed in a platinum capsule with a piece of zinc, Sb, if present, will give a black adherent stain. This may be confirmed by washing the stain with water, then dissolving it in a few drops of hot tartaric acid and a drop or two of HCl; on adding  $\text{H}_2\text{S}$  an orange precipitate ( $\text{Sb}_2\text{S}_3$ ) is formed.

Test I. may also yield non-volatile coatings of Sn or Zn near the assay, yellow hot and white cold; yellow coatings of Pb or Bi; crystalline yellow and white coating of Mo; brick-red to crimson coating and horseradish odor of Se; and deep brown coatings of Cd and Te. All of these will be detected with greater certainty by later tests.

TEST II.—Mix a portion with a spoonful of soda and a little borax and heat strongly upon charcoal with R. F. for three or four minutes. Preserve the fused mass for subsequent examination.

**As.**—Garlic odor and a white volatile coat.

**Cd.**—Dark brown volatile coat, sometimes shading to greenish yellow, and usually surrounded by a variegated coloration resembling the colors of peacock feathers.

*Confirmation Cd.*—The coat forms at first heating, and, if mixed with  $\text{Na}_2\text{S}_2\text{O}_3$  and fused in a borax bead, will form a bright yellow mass of CdS.

**Zn.**—White not easily volatile coat, yellow when hot.

**Sn.**—White non-volatile coat close to assay, yellow while hot and usually small in amount.

*Confirmation Zn and Sn.*—If any coat forms, moisten it with cobalt solution and blow a strong blue flame on the substance. The coatings from other elements will not prevent the cobalt coloration. The zinc coat is made bright yellowish green. The tin coat becomes bluish green.

Test II. may also yield white coats from Pb, Bi, or alkalis, yellow coats from Pb or Bi, brown or red coats from Cu or Mo, and the ash of the coal may be white or red.

**TEST III.**—Crush and pulverize the soda of Test II.; collect any magnetic particles with the magnet; dissolve some of the magnetic particles in a borax bead with the O. F. Try also effect of R. F.; then also place some of the non-magnetic portion of the powder upon a bright silver surface; moisten with water and let stand.\*

**Fe.**—The bead is: O. F. hot, yellow to red; O. F. cold, colorless to yellow; R. F. cold, bottle-green.

*Confirmation Fe.*—The magnetic particles yield with  $\text{HNO}_3$  a brown solution from which, after evaporating excess of acid,  $\text{K}_4\text{FeCy}_6$  throws down a blue precipitate.

**Ni.**—The bead is: O. F. hot, intense violet; O. F. cold, pale brown; R. F. cold, colorless.

*Confirmation Ni.*—If the excess of acid is driven off by evaporation, KCy added in excess, and the solution then made strongly alkaline with KOH, two or three drops of pure bromine will give a black precipitate of  $\text{Ni}_2(\text{OH})_6$ .

\* There may be contained in the soda also metallic particles or buttons, which may be roughly recognized by color and tenacity, as follows:

*Malleable.*—Ag, silver-white; Sn, white; Pb, gray; Au, yellow; Cu, red.

*Brittle.*—Sb, white; Bi, reddish white.

**Co.**—The bead is: O. F. and R. F. hot or cold, a deep pure blue; if greenish when hot, probably Fe or Ni is also present.

*Confirmation Co.*—The magnetic particles yield with  $\text{HNO}_3$  a rose-red solution which becomes blue on evaporation.

**S, Se, Te.**—The bright silver is stained black or dark brown, and unless the horseradish odor of Se or the brown coatings of Se and Te with bismuth flux have been already obtained, this stain will prove sulphur.

*Confirmation S.*—The soda fusion will evolve  $\text{H}_2\text{S}$  when moistened with  $\text{HCl}$ . By holding in the gas a piece of filter-paper moistened with a drop or two of lead acetate (test is made more sensitive by adding a drop of ammonia to the acetate) the paper will be stained black.

*Confirmation Se.*—Characteristic disagreeable horseradish odor during fusion.

*Confirmation Te.*—If a little of the original substance is dropped into boiling concentrated  $\text{H}_2\text{SO}_4$ , a deep violet color is produced; this disappears on further heating.

The quite cold soda fusion added to hot water produces a purple-red solution.

**TEST IV.**—Mix a portion of the substance with more than an equal volume of bismuth flux,\* and heat gently upon a plaster tablet with the oxidizing flame.

**Pb.**—Chrome-yellow coat, darker hot, often covering the entire tablet.

*Confirmation Pb.*—If the test is made on charcoal, the coat is greenish yellow, brown near the assay.

**Hg.**—Gently heated, bright scarlet coat, very volatile,

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\* Formed by grinding together 1 part KI, 1 part  $\text{KHSO}_4$ , 2 parts S.

and with yellow fringe ; but if quickly heated, the coat formed is pale yellow and black.

*Confirmation Hg.*—If the substance is heated gently in a closed tube or matrass with dry soda or litharge, a mirror-like sublimate will form, which may be collected into little globules of Hg by rubbing with a match end. The test with bismuth flux on charcoal yields only a faint yellow coat.

**Bi.** — Bright chocolate-brown coat, with sometimes a reddish fringe.

*Confirmation Bi.*—The coat is turned orange-yellow, then cherry-red, by fumes of  $\text{NH}_3$ , which may conveniently be produced by heating a few crystals of S. Ph. on the assay. The test with bismuth flux on charcoal yields a bright red band, with sometimes an inner fringe of yellow.

**Sb.**—Orange to peach-red coat, very dark when hot.

*Confirmation Sb.*—The coat becomes orange when moistened with  $(\text{NH}_4)_2\text{S}$ .

Test IV. may yield colored sublimates with large amounts of certain other elements, and on smoked plaster certain white sublimates are obtainable. In all cases the elements are detected with greater certainty by other tests, but for convenience they are here summarized : Sn, brownish orange ; As, reddish orange ; Se, reddish brown ; Te, purplish brown, with deep brown border ; Mo, deep ultramarine blue ; Cu, Cd, Zn, white on smoked plaster.

**TEST V.**—Dissolve substance in salt of phosphorus in O. F. so long as bead remains clear on cooling. Treat then for three or four minutes in a strong R. F. to remove volatile compounds. Note the colors hot and cold, then re-oxidize and note colors hot and cold.

**Fe, Ti, Mo, W.**—The bead in O. F. cold is *colorless* or very *faint yellow*.

*Confirmation Fe.*—The bead in its previous treatment should have been O. F. hot, yellow to red; O. F. cold, colorless; R. F. cold, bottle-green.

*Confirmation Ti.*—The bead is reduced on charcoal with tin, pulverized, and dissolved in HCl (1 to 4) with a little metallic tin. The reduced bead is violet, the solution is violet and turbid.

*Confirmation Mo.*—Tested as above on charcoal with tin, etc., the reduced bead is green, the solution is dark brown. Heat a little of the substance on platinum-foil with a few drops of conc.  $\text{HNO}_3$ , heat until excess of  $\text{HNO}_3$  has all volatilized, then add a few drops of strong  $\text{H}_2\text{SO}_4$  and heat until copious fumes are evolved; cool, and breathe upon the cooled mass; an ultramarine blue = Mo.

*Confirmation W.*—Tested on charcoal with tin, etc., as above, the reduced bead is green, the solution is deep blue.

**U, V, Ni.\***—The bead in O. F. cold is colored *yellow* or *greenish yellow*.

\* If the absence of Ni is not proved, or Co obscures the tests, dissolve the substance in borax on charcoal to saturation, and treat for five minutes in hot R. F.

If a visible button results, separate it from the borax, and treat with S. Ph. in the O. F., replacing the S. Ph. when a color is obtained.

If no visible button results, add either a small gold button or a few grains of test-lead. Continue the reduction, and, if lead has been used, scorify the button with frequently changed boracic acid to small size, stopping the instant the boracic acid is colored by Co, Ni, or Cu, blue, yellow, or red, respectively.

Complete the removal of lead by O. F. on coal, and treat as below.

Treat the gold alloy, or the residual button from the lead alloy, on coal, with frequently changed S. Ph., in strong O. F.

The metals which have united with the gold or lead, will be successively oxidized, and their oxides will color the S. Ph. in the following order :

*Confirmation U.*—The bead in R. F. is dull green hot, fine green cold. Make a  $\text{Na}_2\text{CO}_3$  fusion, dissolve in  $\text{HCl}$  or  $\text{H}_2\text{SO}_4$ , add a few drops of  $\text{H}_2\text{S}$  water, and if it gives any precipitate, add it in excess and filter; to filtrate add a few drops of  $\text{HNO}_3$  and boil, then add  $\text{NH}_4\text{OH}$  to alkaline reaction, filter, wash precipitate with ammonia water, and then treat precipitate with a concentrated solution of  $(\text{NH}_4)_2\text{CO}_3 + \text{NH}_4\text{OH}$ , filter, acidify filtrate with  $\text{HCl}$ , and add  $\text{K}_4\text{FeCy}_6$ . Brown ppt. shows U.

*Confirmation V.*—In R. F. the bead will be brownish hot, fine green cold. Fuse substance with  $\text{Na}_2\text{CO}_3$  in O. F., and dissolve fusion in a few drops of dilute  $\text{H}_2\text{SO}_4$  or  $\text{HCl}$ , add a piece of zinc and warm; blue color changing to green and finally violet = V.

*Confirmation Ni.*—The hot bead in O. F. will be intense violet, and in R. F. will be reddish hot, yellow cold.

*Mn.*—The bead in O. F. cold is colored *violet*; if touched while hot to a crystal of nitre, it is made deep permanganate color.

*Confirmation Mn.*—Fused on platinum wire in O. F. with a paste of soda and nitre, manganese yields an opaque bluish-green bead.

*Cr.*—The bead in O. F. cold is colored *green*.

There may be a green bead from admixture of a blue and a yellow. If Cr is not proved, examine in such a case for U, V, Cr, etc., with unusual care.

Co.—Blue, hot; blue, cold. May stay in the slag.

Ni.—Brown, hot; yellow, cold. May give green with Co or Cu.

Cu.—Green, hot; blue, cold. Made opaque red by tin and R. F.

The slag should contain the more easily oxidizable metals, and be free from Cu, Ni, and Ag. Test a portion with S. Ph and tin to prove absence of Cu. If present, it must be removed by further reduction with lead. Pulverize the slags and dissolve a portion in S. Ph., and examine by Test V.

*Confirmation Cr.*—If the substance is fused on platinum wire in the O. F. with a paste of soda and nitre, an opaque yellow bead is produced ; and if the soda bead is dissolved in water, filtered, acidified with acetic acid and a drop or two of lead acetate added, a yellow precipitate will be formed.

*Co, Cu.*—The bead in O. F. cold is colored *blue*.

*Confirmation Co.*—The bead is deep blue, hot and cold, in both flames.

*Confirmation Cu.*—The bead is green hot, greenish blue cold, and on fusion with tin on coal becomes opaque brownish red.

With larger percentage of copper the substance will yield a mixed azure-blue and green flame on heating with HCl.

*SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, SnO<sub>2</sub>.*—The saturated bead contains an appreciable amount of *insoluble material*, in the form of a *translucent* cloud, jelly-like mass, or skeleton form of the original material.

*Confirmation SiO<sub>2</sub>.*—Mix the *dry* substance with a little *dry* calcium fluoride free from SiO<sub>2</sub>, place in a *dry* test-tube and add conc. H<sub>2</sub>SO<sub>4</sub> and heat gently, hold in fumes given off a drop of water in loop of platinum wire ; SiO<sub>2</sub> will be separated on coming in contact with the water and form a jelly-like mass.

Silica or silicates fused with soda unite with noticeable effervescence.

*Confirmation Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, SnO<sub>2</sub>, SiO<sub>2</sub>.*—If infusible, moisten the pulverized mineral with dilute cobalt nitrate solution and heat strongly.

*Al<sub>2</sub>O<sub>3</sub>.*—Beautiful bright blue.

*TiO<sub>2</sub>.*—Yellowish green.



$\text{SnO}_2$ .—Bluish green.

$\text{SiO}_2$ .—Faint blue ; deep blue if fusible.

There may also be blues from fusible phosphates and borates, greens from oxides of Zn, Sb, violet from Zr, various indefinite browns and grays, and a very characteristic pale pink or flesh color from Mg.

*Confirmation  $\text{SnO}_2$ .*—Treat the finely pulverized mineral with Zn and HCl in contact with platinum. Dissolve any reduced metal in HCl and test with  $\text{HgCl}_2$ . There will be white or gray ppt.

**Ba, Ca, Sr, Mg.**—The saturated bead is *white* and *opaque*, and the nearly saturated bead can be flamed white and opaque.

*Confirmation Ba, Ca, Sr.*—Moisten the flattened end of a clean platinum wire with dilute hydrochloric acid, dip it in the roasted substance, and heat strongly at the tip of the blue flame, and gently near the wick. Remoisten with the acid frequently.

**Ba.**—Yellowish-green flame, bluish green through green glass.

**Ca.**—Yellowish-red (brick-red) flame, green through green glass.

**Sr.**—Scarlet-red flame, faint yellow through green glass.

There may also be produced Li, carmine-red flame, invisible through green glass ; K, rose-violet flame, reddish violet through blue glass ; Na, orange-yellow flame, invisible through blue glass ; Cu, azure-blue and emerald-green ; Se and As, pale blue ; Mo, Sb, Te, pale green.

*Confirmation Mg.*—Moisten the roasted substance with cobalt solution and heat strongly. The substance will be colored pale pink or flesh color, or violet if present as either arsenate or phosphate.

**TEST VI.**—Cupellation for silver and gold. Fuse 1 vol. of the roasted substance on charcoal with 1 vol. of borax glass and 1 to 2 vols. of test-lead in R. F. for about two minutes. Remove button and scorify it in R. F. with fresh borax, then place button on cupel and blow O. F. *across* it, using as strong blast and as little flame as are consistent with keeping the button melted. If the litharge is dark, or if the button freezes before brightening, or if it brightens but is not spherical, rescorify it on charcoal with borax, add more test-lead, and again cupel until there remains only a bright spherical button unaltered by further blowing.

**Ag.**—The button is white.

**Au.**—The button is yellow or white.

*Confirmation Ag and Au.*—Dissolve in a drop of  $\text{HNO}_3$ , and add a drop of  $\text{HCl}$ , producing a white curd-like precipitate. If gold is present, there will be a residue insoluble in  $\text{HNO}_3$  which will become golden yellow on ignition.

**TEST VII.**—Heat substance in matrass with acid potassium sulphate.

**$\text{N}_2\text{O}_5$ , Br.**—Reddish-brown vapor.

*Confirmation  $\text{N}_2\text{O}_5$ .*—The gas turns ferrous sulphate paper brown. Nitrates deflagrate violently when fused on charcoal.

**Cl.**—Colorless or yellowish-green vapor, with odor of chlorine.

**I.**—Violet choking vapor.

*Confirmation Br, Cl, I.*—Saturate a salt of phosphorus bead with  $\text{CuO}$ , add substance, and treat in O. F. Br, azure-blue and emerald-green flame. Cl, azure-blue flame with a little green. I, emerald-green flame.

Fuse with  $\text{Na}_2\text{CO}_3$ , pulverize and mix with  $\text{MnO}_2$ , and add a few drops of concentrated  $\text{H}_2\text{SO}_4$ , and heat. Cl, yellowish-green gas that bleaches vegetable colors. Br, red fumes.

Fuse with  $\text{Na}_2\text{CO}_3$ , dissolve in water, make slightly acid with  $\text{H}_2\text{SO}_4$ , and add  $\text{Fe}_2(\text{SO}_4)_3$  (ferric alum may be used), and boil. I, violet fumes (turn starch-paper blue).

**F.**—The glass of the matrass is corroded, and if  $\text{SiO}_2$  is present a film of  $\text{SiO}_2$  is often deposited on the glass.

*Confirmation F.*—If the substance be mixed with silica and then heated with concentrated sulphuric acid, and the fumes caught on a drop of water held in a loop of platinum wire, gelatinous silica will form in the water.

**TEST VIII.**—Heat the substance gently with water to remove air-bubbles, and then with dilute hydrochloric acid.

**$\text{CO}_2$ .**—Effervescence continuing after heat is removed.

$\text{H}_2\text{S}$ , Cl, and H are sometimes evolved, but usually the odor will distinguish these.

*Confirmation  $\text{CO}_2$ .*—If the gas is passed into lime-water, a white cloud and precipitate will be produced.

**TEST IX.**—Place a piece of Mg wire in a closed tube, and cover the wire with a mixture of soda and the substance. Heat till the mass takes fire, cool, and add water.

**P.**—Evolution of phosphine, recognized by odor.

*Confirmation P.*—Fuse a little of the substance, previously roasted if it contains As, with two or three parts  $\text{Na}_2\text{CO}_3$  and one of  $\text{NaNO}_3$ , dissolve in  $\text{HNO}_3$ , and add excess of  $(\text{NH}_4)_2\text{MoO}_4$ ; yellow ppt. shows  $\text{P}_2\text{O}_5$ . In presence of  $\text{SiO}_2$  it is well to confirm this ppt. by dissolving it in dilute  $\text{NH}_4\text{OH}$ , allowing it to stand for half an hour and filtering off any  $\text{SiO}_2$  that separates, then to filtrate add-

ing magnesia mixture ( $\text{MgCl}_2 + \text{NH}_4\text{Cl} + \text{NH}_4\text{OH}$ ); white crystalline ppt. shows  $\text{P}_2\text{O}_5$ .

Phosphates yield a pale momentary bluish-green flame when moistened with concentrated  $\text{H}_2\text{SO}_4$  and treated at the tip of the blue flame.

TEST X.—Make a paste of four parts  $\text{KHSO}_4$ , one part  $\text{CaF}_2$ , water, and substance. Treat at tip of blue flame. Just after water is driven off the flame will be colored.

B.—Bright green.

Li.—Carmine.

*Confirmation B.*—Heat some of the substance gently on platinum wire, then add a drop of concentrated  $\text{H}_2\text{SO}_4$ , heat very gently again, just enough to drive off excess of  $\text{H}_2\text{SO}_4$ , dip in glycerine, hold in flame until glycerine begins to burn, remove from flame, and the mass will continue burning with a green flame.

SPECIAL SCHEME FOR DETECTION OF THOSE METALS  
WHICH WHEN PRESENT AS SILICATES USUALLY FAIL  
TO YIELD SATISFACTORY TESTS BEFORE THE BLOW-  
PIPE.

Remove the volatile constituents as thoroughly as possible by roasting, then heat gently in a platinum capsule with HF and a few drops of concentrated  $\text{H}_2\text{SO}_4$  as long as fumes are given off; add a little more HF and  $\text{H}_2\text{SO}_4$ , and heat again in the same way. When fusion is quite cold, dissolve in cold water and filter.

FILTRATE *a*.—Divide into four parts and test as follows:

1. Add a piece of Zn or Sn and a little HCl, and heat.

Ti.—A violet or blue solution.

*Confirmation Ti.* — Nearly neutralize solution, and then add  $\text{Na}_2\text{S}_2\text{O}_8$ , and boil. White ppt. shows Ti.

Or, make solution slightly alkaline, and then acidify slightly with  $\text{HCl}$ , and add  $\text{Na}_2\text{HPO}_4$ . White ppt. shows Ti.

2. Add excess of  $\text{KOH}$  or  $\text{NaOH}$ , boil and filter, and to filtrate add excess of  $\text{NH}_4\text{Cl}$ , and boil.

**Al.**—White precipitate.

Dissolve ppt., produced by the  $\text{KOH}$  or  $\text{NaOH}$ , in  $\text{HCl}$ , and add  $\text{K}_4\text{FeCy}_6$ .

**Fe.**—Blue precipitate.

3. Add  $\text{HCl}$ ; then make alkaline with  $\text{NH}_4\text{OH}$  and add  $(\text{NH}_4)_2\text{S} + (\text{NH}_4)_2\text{CO}_3$  in slight excess; filter; to filtrate add  $\text{Na}_2\text{HPO}_4$ .

**Mg.**—White *crystalline* precipitate.

*Confirmation Mg.*—If phosphates are present, this test would not be reliable for Mg. In such cases test a few drops of the solution with  $\text{H}_2\text{S}$ ; if it causes any precipitate, saturate the whole of the solution with it, filter, and to filtrate add a few drops of  $\text{HNO}_3$ , and boil to oxidize  $\text{FeO}$ , nearly neutralize with solution of  $\text{Na}_2\text{CO}_3$ . If iron is not present, add a few drops of  $\text{Fe}_2\text{Cl}_6$ , enough to give a red precipitate with the sodium acetate, then dilute and add excess of sodium acetate, and boil, filter, and to filtrate add  $\text{NH}_4\text{OH} + (\text{NH}_4)_2\text{S}$ , filter, to filtrate add  $\text{Na}_2\text{HPO}_4$ . White crystalline precipitate shows Mg.

4. Add  $\text{BaCl}_2$  as long as it gives a precipitate, then  $\text{Ba}(\text{OH})_2$  to alkaline reaction, boil, filter, and to filtrate add  $(\text{NH}_4)_2\text{CO}_3$  and  $\text{NH}_4\text{OH}$  and heat; filter; evaporate filtrate to dryness and ignite to drive out  $\text{NH}_4$  salts. Test residue in flame for K and Na; dissolve residue in a few drops of water, filter if necessary, and then add solution of  $\text{H}_2\text{PtCl}_6$  and alcohol.

**K.**—Yellow crystalline precipitate.

*Confirmation Na, K.*—Mix 1 part of the silicate with from 5 to 6 parts of precipitated  $\text{CaCO}_3$  and 1 part of  $\text{NH}_4\text{Cl}$ , heat to redness in platinum capsule for thirty minutes, being careful to apply heat gently at first, digest sintered mass in hot water, and filter; to filtrate add  $(\text{NH}_4)_2\text{CO}_3$  and  $\text{NH}_4\text{OH}$ , heat, and filter; evaporate filtrate to dryness and ignite gently until all ammonium salts are driven off, then determine Na and K as above.

**RESIDUE a.**—Boil with strong solution of  $(\text{NH}_4)_2\text{SO}_4$  and filter.

**FILTRATE b.**—Add a few drops of  $\text{H}_2\text{S}$  water; if any precipitate forms, saturate with  $\text{H}_2\text{S}$  and filter, and to filtrate add  $\text{NH}_4\text{OH}$  and  $(\text{NH}_4)_2\text{C}_2\text{O}_4$ .

**Ca.**—A white precipitate.

**RESIDUE b.**—Moisten with concentrated  $\text{HCl}$  and try coloration of flame.

**Ba.**—Yellowish-green flame.

**Sr.**—Scarlet flame.

*Confirmation Ba and Sr.*—Fuse residue *b* with two to three pts. of soda in a platinum capsule; treat fusion with boiling water, filter, reject filtrate, dissolve residue in acetic acid, add a few drops of  $\text{H}_2\text{S}$  water, if it gives any precipitate, saturate with  $\text{H}_2\text{S}$  and filter, and to filtrate add  $\text{K}_2\text{Cr}_2\text{O}_7$ . Ba gives a yellow precipitate. Filter, and to filtrate add  $\text{CaSO}_4$ , warm, and let stand. Sr gives a white precipitate.



$\text{As}_2\text{S}_3$ .  $\text{PtS}_2$ .  $\text{Sb}_2\text{S}_3$ .  $\text{SnS}$ .  
thoroughly with  $\text{H}_2\text{O}$ .

*Filtrate 7*

$\text{H}_4\text{AsS}_3$ .  $(\text{NH}_4)_3\text{SbS}_4$ .  $(\text{NH}_4)_3\text{AsS}_3$ .  $(\text{NH}_4)_2\text{SnS}_2$ .  $(\text{NH}_4)_2\text{PtS}_2$ .  
Precipitate by adding  $\text{HCl}$  to acid reaction, filter, and wash with  $\text{H}_2\text{O}$ .

*Precipitate I.*

In  $\text{HCl}$  and very little  $\text{KClO}_3$ , boiling out all  $\text{Cl}$  carefully, add-  
prevent the concentration of solution. Divide into two parts.

*Filtrate.*

Reject.

| 1  |                                 | 2   |                                       |
|--|---------------------------------|---|---------------------------------------|
| e into a small flask with $\text{Zn}$ and $\text{H}_2\text{SO}_4$ ;<br>as into $\text{AgNO}_3$ and filter. |                                 | Add $\text{KCl}$ and evaporate nearly<br>to dryness over a water-bath;<br>moisten with alcohol and a little<br>$\text{H}_2\text{O}$ , and filter. |                                       |
| te J.  | <i>Filtrate 9.</i>              | <i>Zn and Precipitate K.</i>  | <i>Precipitate L.</i>                 |
| with   | Add $\text{AgNO}_3$ and         | (From the flask.)   | Red or yellow                         |
| add  | carefully neu-                  | Detach ppt from   | ppt = $2\text{KCl}$ , $\text{PtCl}_4$ |
| filter;  | tralize with very               | the $\text{Zn}$ , transfer  | = $\text{Pt}$ .                       |
|  | dilute $\text{NH}_4\text{OH}$ . | to test-tube; dis-  |                                       |
| e 8.   | A yellow ppt =                  | solve in $\text{HCl}$ and   |                                       |
| 8 gas.   | $\text{Ag}_3\text{AsO}_3$ . Or  | add $\text{HgCl}_2$ .   |                                       |
| ppt =  | ppt $\text{AgNO}_3$ by          | A white or gray   |                                       |
|  | $\text{HCl}$ and filter.        |   |                                       |
|  |                                 |   | <i>Filtrate 10.</i>                   |
|  |                                 |   | Add $\text{H}_3\text{C}_2\text{O}_4$  |
|  |                                 |   | and warm.                             |
|  |                                 |   | Ppt = $\text{Au}$ .                   |







## TABLE OF QUALITATIVE

## PART II—AC

Boil the mixture containing the acids with

| <i>Precipitate A.</i>  |  |   |                    |
|--|--|---|--------------------|
| Contains bases, and perhaps Silicates, Phosphates, and Fluorides. Divide into two parts.   |  |   |                    |
| <i>Part 1.</i>   |  | <i>Part 2.</i>  |                    |
| Acidify with $\text{HNO}_3$ and evaporate to dryness. Take up residue with $\text{HNO}_3$ and $\text{H}_2\text{O}$ , filter, and wash. |  | Acidify with $\text{HC}_2\text{H}_3\text{O}_2$ and filter.  |                    |
| <i>Residue B.</i>  | <i>Filtrate 1.</i>   | <i>Residue C.</i>   | <i>Filtrate 2.</i> |
| A white gritty powder = $\text{SiO}_2$ .<br>Test with metaphosphate bead.  | Add $(\text{NH}_4)_2\text{MoO}_4$ . A yellow crystalline ppt = $\text{H}_2\text{PO}_4$ . | If no $\text{SiO}_2$ has been found in Residue B, treat Residue C with $\text{H}_2\text{SO}_4$ (conc.) in a Pb or Pt dish, and see if fumes will etch; if they do = HF. In the presence of $\text{SiO}_2$ , add $\text{H}_2\text{SO}_4$ and distil from test-tube into tube of water. Water becomes acid ( $\text{H}_2\text{SiF}_6$ ) and free silica separates indicates HF. | Reject             |

Group 1.

If Group 1 has been found to be present by test with  $\text{BaCl}_2$  on Part 1 ( $\frac{3}{4}$  of Filtrate 5), take a third part of this solution

| <i>Precipitate D.</i>  |   |  |   |
|--|---|--|---|
| Treat with $\text{HC}_2\text{H}_3\text{O}_2$ and filter.   |   |  |   |
| <i>Residue E.</i>  |   | <i>Filtrate 2.</i>   |   |
| Test a portion in a metaphosphate bead for $\text{SiO}_2$ . Divide the rest into two parts.  |   | Divide into two parts.   |   |
| <i>Part 1.</i>   | <i>Part 2.</i>  | <i>Part 1.</i>   | <i>Part 2.</i>  |
| Dry, ignite gently, then add $\text{HC}_2\text{H}_3\text{O}_2$ . Effervescence = $\text{CO}_2$ = $\text{H}_2\text{C}_2\text{O}_4$ .<br>Or acidify Sol. (a) with $\text{HC}_2\text{H}_3\text{O}_2$ , add $\text{CaSO}_4$ . A white pulverulent ppt = $\text{CaC}_2\text{O}_4$ . | If $\text{SiO}_2$ is not present, heat in Pb or Pt dish with $\text{H}_2\text{SO}_4$ (conc.) for HF (etching test). | Acidify slightly with $\text{HCl}$ and test for $\text{H}_2\text{BO}_3$ with turmeric paper. Pink color.             | Acidify with $\text{HCl}$ and test for $\text{H}_2\text{BO}_3$ with turmeric paper. Pink color. |
|  |   | Test original solution with $\text{HCl}$ ; if $\text{HC}_2\text{H}_3\text{O}_2$ with effervescence = $\text{CO}_2$ . |   |

Group 2.

Divide into two parts.

| <i>Part 1.</i>   | <i>Precipitate.</i>  |
|--|--|
| Add a little $\text{CS}_2$ and then a few drops of $\text{HCl}$ , then $\text{NaClO}$ very carefully, the I is set free and colors $\text{CS}_2$ purple; add more $\text{NaClO}$ very carefully, and finally the I color disappears and the Br is then liberated and colors $\text{CS}_2$ brown. | $\text{H}_2\text{S}$ , $\text{HCN}$ , $\text{H}_2\text{Fe}(\text{CN})_6$ , $\text{H}_2\text{Fe}(\text{CN})_5$ , $\text{HCl}$ , $\text{HI}$ , and $\text{HBr}$ . Boil out $\text{H}_2\text{S}$ , test fumes and pass distillate into $\text{KOH}$ ; test this for $\text{HCy}$ by adding $\text{FeSO}_4$ , $\text{KOH}$ , $\text{Fe}_2\text{C}_6$ , and formed, and until the liquid becomes blue or green; filter. |
|  | $\text{Cu}_2\text{Fe}(\text{CN})_6$ , $\text{Cu}_2\text{Fe}_2(\text{CN})_{10}$ .   |
|  | NOTE.—Test for these acids in the original with Ferric and Ferrous salts.  |
|  | Acidify with $\text{HCl}$ and test with $\text{FeSO}_4$ ; a blue ppt = $\text{Fe}_2(\text{CN})_{12}$ . The $\text{FeSO}_4$ must be recently made.  |
|  | Acidify with $\text{HCl}$ and add $\text{Fe}_2\text{Cl}_6$ ; a blue ppt = $\text{Fe}_7(\text{CN})_{18}$ .  |

this is dissolved

ORGANIC ACIDS.  $\text{HC}_2\text{H}_3\text{O}_2$ .  
Use the original neutral solution.

| <i>Part 1.</i>  | <i>Part 2.</i>   |
|---|--|
| For $\text{HC}_2\text{H}_3\text{O}_2$ add conc. $\text{H}_2\text{SO}_4$ and $\text{C}_2\text{H}_5\text{OH}$ , heat gently; odor of ethyl acetate (cider). | For $\text{H}_2\text{C}_2\text{H}_3\text{O}_4$ add one drop of $\text{FeSO}_4$ , $\text{NaClO}$ and excess of $\text{KOH}$ . Purple color. |

# ATIVE ANALYSIS.

## -ACIDS.

with excess of  $\text{Na}_2\text{CO}_3$ , filter hot.

### Filtrate 5.

Contains the acids and sodium salts of As. Divide into two unequal parts.

#### Part 1 ( $\frac{3}{4}$ of Filtrate 5).

Add  $\text{HNO}_3$  to faintly acid reaction, then  $\text{NH}_4\text{OH}$  to slightly alkaline reaction, and boil until all free  $\text{NH}_4\text{OH}$  is driven off. Divide into two parts.

#### Part 1.

Take a small portion of this neutral solution and add  $\text{BaCl}_2$ . A white ppt = Acids of Group 1. To this ppt add  $\text{HCl}$ ; all dissolve except  $\text{BaSO}_4$  and  $\text{BaSiF}_6$ . The latter need not be looked for in the mixture.

#### Part 2.

Take a small portion of this neutral solution, add  $\text{AgNO}_3$ , and then acidify with  $\text{HNO}_3$ . Ppt = Acids of Group 2. Note the color of the ppt before and after adding  $\text{HNO}_3$ .

#### Part 2 ( $\frac{1}{4}$ of Filtrate 5).

Add to a portion of this solution an equal volume  $\text{H}_2\text{SO}_4$  (conc.) and few drops of  $\text{FeSO}_4$ . Brown ring ( $\text{FeSO}_4 \cdot \text{NO}$ ; in presence of  $\text{HClO}_4$ ,  $\text{HBr}$ ,  $\text{HI}$ ,  $\text{H}_2\text{CrO}_4$ , add  $\text{Zn}$  and  $\text{KOH}$  and boil. Odor of  $\text{NH}_3 = \text{HNO}_3$ . Test second portion for  $\text{HClO}_4$  by adding  $\text{Ag}_2\text{SO}_4$ , filter, reject ppt, evaporate solution to dryness, take up with  $\text{H}_2\text{O}$ , and test for chlorides.

o 1.

tion and test as follows: Add  $\text{CaCl}_2$  and enough  $\text{NH}_4\text{OH}$  to make the solution slightly alkaline, warm, and filter.

### Filtrate 3.

Add  $\text{BaCl}_2$ . Ppt =  $\text{BaSO}_4$  and  $\text{BaCrO}_4$ . Filter.

te 2.

wo parts.

### Precipitate F.

Treat with  $\text{HCl}$  and filter.

#### Part 2.

Acidify with  $\text{HNO}_3$  and test for  $\text{P}_2\text{O}_5$  with  $(\text{NH}_4)_2\text{MoO}_4$ . A yellow ppt in cold solution =  $\text{P}_2\text{O}_5$ .

#### Residue G.

=  $\text{BaSO}_4$ . Fuse on charcoal with  $\text{Na}_2\text{CO}_3$  and test on  $\text{Ag}$ . Brown stain  $\text{Ag}_2\text{S}$ .

#### Filtrate 4.

Add  $\text{NH}_4\text{OH}$  to alkaline reaction. Yellow ppt =  $\text{BaCrO}_4$ .

### $\text{CO}_2$ .

if effervescence, pass the gas into limewater. A white ppt soluble in

o 2.

wo parts.

#### Part 2.

fumes with  $\text{PbA}$  paper; a black coloration indicates  $\text{H}_2\text{S}$ . Add  $\text{NaHCO}_3$  and distil, carrying distillation to dryness, and  $\text{HCl}$ ; blue ppt. When cyanides are removed, cool, dissolve residue in  $\text{H}_2\text{O}$  and add  $\text{CuSO}_4$  as long as a ppt is

### Solution.

Add conc. solution of  $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2$  and boil, pass vapor into starch-water. Blue color indicates  $\text{HI}$ . When this is removed, add a drop of conc.  $\text{H}_2\text{SO}_4$ , some dil.  $\text{K}_2\text{Mn}_2\text{O}_8$ , and boil as long as  $\text{Br}$  is given off, or until the color is discharged, adding more  $\text{K}_2\text{Mn}_2\text{O}_8$  if necessary. Finally test solution with  $\text{HNO}_3$  and  $\text{AgNO}_3$ ; a white curdy ppt soluble in  $\text{NH}_4\text{OH}$  and pptd by  $\text{HNO}_3$  indicates  $\text{HCl}$ .

o 2.  $\text{H}_3\text{C}_4\text{H}_4\text{O}_6$ .  $\text{H}_3\text{C}_5\text{H}_8\text{O}_7$ .

ion. Divide into three parts.

2.

$\text{O}_2$  and one or two drops of  $\text{H}_2\text{O}_2$ , or or.

#### Part 3.

For  $\text{H}_3\text{C}_5\text{H}_8\text{O}_7$ , add  $\text{CaCl}_2$ , filter cold, reject the ppt of  $\text{CaC}_4\text{H}_4\text{O}_6$ , boil the filtrate. The ppt is  $\text{Ca}_3(\text{C}_5\text{H}_8\text{O}_7)_2$ .





# ZETTNOW'S SCHEME FOR QUALITATIVE ANALYSIS

ARRANGED BY H. C. J.

For the Students of the School of Chemistry

Add hydrochloric acid to the solution, wash, and filter.

| <i>Precipitate.</i><br>Boil with water and filter. |  |  | <i>Filtrate.</i><br>Add excess of dilute $H_2SO_4$ and wash on filter.   |  |  |
|--|--|--|--|--|--|
| <i>Solution.</i><br>Add $H_2SO_4$                  | <i>Residue.</i><br>Treat with $(NH_4)HO$ |  | <i>Precipitate.</i><br>Agitate with considerable cold water and filter.  |  | <i>Filtrate.</i><br>Divide the solution into two   |
| <i>Precipitate</i><br><b>Pb</b>                    | <i>Solution.</i><br>Add $HNO_3$          | <i>Residue</i><br>turns gray or black<br><b>Hg</b> | <i>Filtrate.</i><br>Add excess of $(NH_4)_2C_2O_4$   | <i>Residue.</i><br>Add $(NH_4)_2C_2O_4$ and digest and filter.   | To $\frac{1}{2}$ add $BaH_2O_3$ and boil.  |
|  | <i>Precipitate</i><br><b>Ag</b>          |  | <i>Precipitate</i><br><b>Ca</b>  |  | Place in   |
|  |  |  | <i>Residue.</i><br>Boil with $Na_2CO_3$ , filter, wash, dissolve on filter with $HCl$ , neutralize filtrate with $(NH_4)HO$ , and divide into two parts. | <i>Filtrate.</i><br>Add $H(C_2H_3O_2)$ and $K_2CrO_4$  | <i>Volatilized</i><br>$(NH_4)_2O$<br>Test gas with $HCl$ and litmus.   |
|  |  |  | <i>Precipitate</i><br><b>Ba</b>  | <i>Precipitate</i><br><b>Pb</b>  | <i>Solution</i><br>Add excess of $(NH_4)_2CO_3$ and $(NH_4)_2C_2O_4$   |
|  |  |  | <i>1st Half.</i><br>Add excess of solution of $SrSO_4$   | <i>Second Half.</i><br>Add excess of $H_2SiF_6$ and alcohol. Shake, filter, dilute with water, expel alcohol by evaporation, add solution of $CaSO_4$ , and after one or two minutes a precipitate | warm, filter, evaporate to dryness, and ignite residue. Test on platinum wire in colorless flame; intense yellow color indicates <b>Na</b> . Violet color seen through blue glass indicates <b>K</b> |
|  |  |  |  | <b>Sr</b>  | <i>Volatilized</i><br>Collected spots cold porcelain, and treat with $NaCl$ . Spot-dissolve; <b>As</b> Spots do not dissolve <b>Sb</b> Test also with $AgNO_3$                                       |

In this scheme regard is had to the following substances in aqueous solution :

- I.  $PbO$ ,  $Ag_2O$ ,  $HgO$
- II.  $CaO$ ,  $BaO$ ,  $SrO$
- III.  $(NH_4)_2O$ ,  $Na_2O$ ,  $K_2O$
- IV.  $As_2O_3$ ,  $As_2O_5$ ,  $Sb_2O_3$ ,  $Sb_2O_5$ ,  $SnO$ ,  $SnO_2$ ,  $Hg_2O$ ,  $CuO$ ,  $CdO$ ,  $Bi_2O_3$
- V.  $FeO$ ,  $Fe_2O_3$ ,  $Cr_2O_3$ ,  $Al_2O_3$
- VI.  $MnO$ ,  $MgO$ ,  $CoO$ ,  $NiO$
- VII.  $ZnO$

N. B. To test for zinc mix filter, add  $NaHO$  in excess, an filtrate, boil until all odor of  $(NH_4)$  solution, a cloud or precipitate

# ANALYSIS WITHOUT THE USE OF H<sub>2</sub>S OR (NH<sub>4</sub>)<sub>2</sub>HS.

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to two unequal parts,  $\frac{1}{2}$  and  $\frac{1}{2}$ .

Place  $\frac{1}{2}$  of the solution in a Marsh's apparatus, add pieces of zinc and a strip of platinum foil, when but little zinc remains heat 15 or 20 minutes, and throw contents of flask on a filter; wash thoroughly.

Tol-  
lized.  
ollect  
spots on  
cold  
porce-  
lain-  
and  
treat  
with  
NaCO<sub>3</sub>.  
Spots  
dis-  
olve.  
As  
spots  
not  
dis-  
olve.  
Sb  
Test  
also  
with  
HNO<sub>3</sub>

*Residue.*  
Treat with strong HNO<sub>3</sub>, and filter.

*Residue.*  
Wash, boil with  
HCl and filter.

*Filtrate.*  
Divide into two parts.

*Solu-  
tion.*  
Put in a plat-  
inum  
dish  
with a  
piece of  
zinc.  
A dark  
spot on  
the plat-  
inum  
indi-  
cates  
**Sb**

*Resi-  
due.*  
Add to solution  
in plat-  
inum  
dish,  
boil  
with  
HCl,  
filter  
and add  
HgCl<sub>2</sub>  
*Preci-  
pitate.*  
**Sn**

*1st Half.*  
Add  
SnCl<sub>2</sub>

*Precip-  
itate.*  
**Hg**

*Second Half.*  
Add HCl, boil, then  
add excess of NaHO,  
wash the precipitate,  
on filter with water,  
then with (NH<sub>4</sub>)HO  
containing NH<sub>4</sub>Cl.

*Residue.*  
Dissolve on  
filter in very  
little HCl  
and add  
large quan-  
tity of H<sub>2</sub>O  
to the fil-  
trate. A  
cloudy pre-  
cipitate in-  
dicates  
**Bi**

*Filtrate.*  
Divide into two  
parts.

*1st Half.*  
Acidify  
with HCl  
and add  
K<sub>4</sub>Fe<sub>2</sub>Cy<sub>6</sub>

*Precip-  
itate.*  
**Cu**

*2d Half.*  
Add  
excess of  
NaHO, a  
white gelat-  
inous  
*Precip-  
itate.*  
**Cd**

*Filtrate*  
Boil with a little HNO<sub>3</sub>, and divide into two un-  
equal parts.

*1st Portion.*  
Add KCyS  
*Red Color*  
**Fe \***

*Second Portion.*  
Neutralize with (NH<sub>4</sub>)HO, add ex-  
cess of Ba CO<sub>3</sub>, agitate ten min-  
utes, filter and wash thoroughly.

*Precipitate.*  
Boil in a porcelain  
dish with dilute  
H<sub>2</sub>SO<sub>4</sub>, and filter.  
Add excess of NaHO  
to filtrate, a few  
drops of KMnO<sub>4</sub>  
and a little NH<sub>4</sub>Cl,  
boil, filter, and  
divide the solution.

*Filtrate.*  
Add excess of dilute H<sub>2</sub>SO<sub>4</sub>,  
filter and saturate filtrate  
with (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub>, warm, fil-  
ter, and wash.

*1st Half.*  
Add some  
H(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)  
and  
Pb(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)<sub>2</sub>  
*Preci-  
pitate*  
**Cr**

*2d Half.*  
Add  
excess of  
NH<sub>4</sub>Cl  
*Preci-  
pitate*  
**A**

*Precip-  
itate.*  
Mix a portion  
with  
Na<sub>2</sub>CO<sub>3</sub>  
and  
NaNO<sub>3</sub>,  
fuse on  
platinum  
foil.  
*Green  
color.*  
**Mn**

*Solution.*  
Add Na<sub>2</sub>HPO<sub>4</sub>

*Preci-  
pitate*  
**Mg**  
*Solu-  
tion.*  
Evapo-  
rate to  
dryness,  
dissolve in HCl,  
add KNO<sub>3</sub> and  
H(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>),  
filter.

\* To determine de-  
gree of oxidation of  
Fe, examine the or-  
iginal solution with  
K<sub>4</sub>Fe<sub>2</sub>Cy<sub>6</sub> and KCyS

Dissolve  
another  
portion in  
HCl, neu-  
tralize  
with  
(NH<sub>4</sub>)HO,  
add con-  
siderable  
NH<sub>4</sub>Cl and  
(NH<sub>4</sub>)<sub>2</sub>C<sub>2</sub>O<sub>4</sub>  
*Precip-  
itate*  
**Ca**

*Preci-  
pitate*  
**Co**

*Solu-  
tion.*  
Add  
Nallo

*Preci-  
pitate*  
**Ni**

mix a portion of the original solution with HCl, H<sub>2</sub>SO<sub>4</sub>,  
and boil. Add a little (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> and NH<sub>4</sub>Cl to fil-  
(NH<sub>4</sub>)HO is expelled, and filter. Add K<sub>4</sub>Fe<sub>2</sub>Cy<sub>6</sub> to  
ate indicates **Zn**.

mix a portion of the original solution with HCl, H<sub>2</sub>SO<sub>4</sub>, and boil. Add a little (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> and NH<sub>4</sub>Cl to filtrate. (NH<sub>4</sub>)HO is expelled, and filter. Add K<sub>4</sub>Fe<sub>2</sub>Cy<sub>6</sub> to indicate **Zn**.





## **PART V.**

### **TABLES, EXAMPLES, AND REFERENCES.**



## PRECIOUS STONES.

### ARRANGED ACCORDING TO HARDNESS.

| NAME.  | COLOR.  | HARD-<br>NESS. | SPECIFIC<br>GRAVITY. | ACTION OF<br>ACIDS.   | BLOWPIPE CHARAC-<br>TERISTICS.                          |
|--|---|----------------|----------------------|---|---|
| Diamond.   | Colorless, smoky,<br>yellow, green,<br>blue, and black. | 10.            | 3.5                  | Not acted on.   | Burns at an intense<br>heat, without<br>residue.        |
| Sapphire<br>(Corundum).                                | Colorless, blue, red,<br>yellow, gray, and<br>brown.    | 9.             | 4                    | Insoluble.  | Infusible.  |
| Topaz.   | Colorless, yellow,<br>blue, greenish-<br>blue.          | 8.             | 3.5                  | Not acted on.   | Infusible, cracks at<br>a high heat.                    |
| Ruby (Spinel).   | Red, blue, green,<br>yellow, white, and<br>black.       | 8.             | 3.5 to 4.5           | Insoluble in hy-<br>drochloric acid.<br>Partly soluble<br>in sulphuric<br>acid. | Infusible, changes<br>color.                            |
| Emerald (Aqua-<br>marine, Beryl).                      | Green, blue, yellow,<br>red, and white.                 | 7.5 to 8       | 2.6 to 2.8           | Not acted on.   | Fuses with difficul-<br>ty on the edges.                |
| Zircon.  | Colorless, yellow,<br>red, brown,<br>pink, and green.   | 7.5            | 4.7                  | Insoluble.  | Infusible.  |
| Agate, Jasper,<br>Amethyst,<br>Onyx, etc.<br>(Quartz). | Colorless, white,<br>black, red, and<br>green.          | 7.             | 2.5 to 2.7           | Insoluble.  | Infusible, except<br>with carbonate of<br>soda.         |
| Garnet.  | Red, brown, yellow,<br>white, green, and<br>black.      | 6.5 to 7.5     | 3.15 to 4.4          | Imperfectly sol-<br>uble.   | Fusible.  |
| Turquoise.   | Blue, white, yellow,<br>and red.                        | 6.             | 2.6 to 2.8           | Soluble.  | Infusible.  |
| Lapis-Lazuli.  | Blue, red, green,<br>and colorless.                     | 5.5 to 5       | 2.4                  | Gelatinizes.  | Fuses with intumes-<br>cence and gives a<br>white bead. |
| Opal.  | Brown, green, and<br>gray.                              | 5.5 to 6.5     | 2.1 to 2.2           | More or less sol-<br>uble.  | Infusible, gives off<br>water and be-<br>comes opaque.  |
| Malachite.   | Bright green.   | 3.5 to 4       | 3.9 to 4             | Soluble with ef-<br>fervescence.  | Gives off water and<br>Fuses.                           |

### SCALE OF HARDNESS.

1. Easily scratched with the nail. Talc.
2. Not easily scratched with the nail; does not scratch a copper coin. Gypsum.

3. Scratches and is scratched by a copper coin. Calcite.
4. Not scratched by a copper coin ; does not scratch glass. Fluorite.
5. Scratches glass with difficulty ; easily scratched with the knife. Apatite.
6. Scratches glass easily ; not easily scratched by the knife. Orthoclase.
7. Not scratched by the knife ; yields with difficulty to the file. Quartz.
8. Harder than flint. Topaz.
9. Harder still. Sapphire.
10. Diamond.

## METALS—CHARACTERISTICS.\*

INCLUDING CARBON AND SULPHUR.

| METAL.    | COLOR.                               | HARDNESS. | SPECIFIC GRAVITY. | BEST SOLVENTS.        | ON CHARCOAL, BEFORE THE BLOWPIPE.            | FUSIBILITY, CENTIGRADE.  |
|-----------|--------------------------------------|-----------|-------------------|-----------------------|--|--------------------------|
| Lead.     | Bluish, malleable.                   | 1.5       | 11.35             | Nitric or Muriatic.   | Fuses and gives a yellow coat.               | 324°                     |
| Antimony. | Bluish-white, brittle.               | 3—3.5     | 6.8               | Aqua regia            | Fuses and gives off white fumes.             | 432°                     |
| Silver.   | White, malleable.                    | 2.5—3     | 10.57             | Nitric and sulphuric. | Fuses, gives reddish coat with long blowing. | 1000°                    |
| Gold      | Yellow, malleable.                   | 2.5—3     | 19.3              | Aqua regia            | Fuses to a button.                           | 1200°                    |
| Platinum. | Whitish to steel-gray, malleable.    | 4—4.5     | 21.48             | Aqua regia            | Infusible.                                   | In flame of oxy. h. blp. |
| Zinc.     | Bluish-white, malleable, brittle.    | 2.        | 6.8—7.2           | All acids.            | Oxidizes and gives a white coat.             | 419°                     |
| Mercury.  | Tin-white, liquid.                   | —1        | 13.59             | Nitric.               | Volatilizes.                                 | Solid at —38.5°          |
| Bismuth.  | Reddish to silver white, brittle.    | 2—3.5     | 9.76              | Nitric.               | Fuses and gives an orange yellow coat.       | 268.3°                   |
| Tin.      | Like silver, more bluish, malleable. | 4—5       | 7.28              | Muriatic. Sulphuric.  | Gives metallic globule and white coat.       | 228°                     |
| Copper.   | Red, malleable.                      | 2.5—3     | 8.9               | nitric.               | Can be fused to a bead.                      | 1100-                    |

\* Specific gravity and melting-points taken from new edition of Watts' Dictionary of Chemistry.

| METALS.    | COLOR.                              | HARDNESS. | SPECIFIC GRAVITY. | BEST SOLVENTS.               | ON CHARCOAL, BEFORE THE BLOWPIPE.    | FUSIBILITY, CENTIGRADE.      |
|------------|-------------------------------------|-----------|-------------------|------------------------------|--------------------------------------|------------------------------|
| Iron.      | Gray, malleable, magnetic.          | 4—5       | 7.8               | All acids.                   | Infusible.                           | Highest heat of Forge. 1600° |
| Manganese. | Grayish-white, brittle.             | 9—10      | 7.39              | Nitric, sulphuric, muriatic. | Infusible.                           | 1800°                        |
| Nickel.    | Silver-white, malleable, magnetic.  | 5—6       | 9                 | Nitric.                      | Infusible.                           | 1400°                        |
| Cobalt.    | Steel-gray to red, magnetic.        | 5—6       | 8.96              | Nitric.                      | Infusible.                           | 1500°                        |
| Carbon.    | Colorless to Black.                 | Variable. | Variable.         | Insoluble.                   | Infusible—burns.                     | Infusible.                   |
| Sulphur.   | Yellow, reddish, greenish, brittle. | 1—2.5     | 2.03              | Oil of Turpentine, etc.      | Melts and gives off sulphurous acid. | 115°                         |

REMARKS.—The value of the various metals changes according to the production and demand, save in the case of gold which has a standard value of twenty dollars and sixty-seven cents per Troy ounce in gold coin, when perfectly pure; the price being determined as follows:

By the laws of the United States the composition of the gold coins for every one hundred parts, by weight, is 90 parts of pure gold and 10 parts of alloy.

Eight hundred dollars in U. S. gold coin weighs 43 ounces Troy,  $\frac{9}{10}$  of this weight must, therefore, be pure gold = 38.7 ounces.  $\frac{800}{38.7} = 20.6718$  dollars; this then is the coin value of one ounce of pure gold.

Before the immense production of silver depreciated the price of that metal, a similar calculation gave its value in gold coin per Troy ounce. No comparison, however, can now be made, the price of silver being variable. The value was deduced as follows:

\$12.80 silver coin = 11 ounces Troy,  $\frac{9}{10}$  of this gives the pure metal = 9.9 ounces.  $\frac{12.80}{9.9} = 1.2929$  as the coin value of 1 ounce of pure silver. Comparing this with the value of 1 ounce of pure gold we have: 1.2929 : 20.6718 :: 1 : 16.

## ORES—CHARACTERISTICS.

## LEAD.

| ORES.         | COMPOSITION.  | COLOR.                         | HARDNESS. | SPECIFIC GRAVITY. | SOLUBILITY. SOLVENTS.      | ON CHARCOAL. BEFORE THE BLOWPIPE.    |
|---------------|---|--------------------------------|-----------|-------------------|----------------------------|--------------------------------------|
| Galena.       | PbS   | Steel-gray.                    | 2.5—2.7   | 7.4—7.7           | Nitric acid.               | Gives off SO <sub>2</sub> and fuses. |
| Cerussite.    | PbCO <sub>3</sub>   | Variable.                      | 3—3.5     | 6.4               | Nitric with effervescence. | Cracks and fuses.                    |
| Anglesite.    | PbSO <sub>4</sub>   | White tinged with yellow, etc. | 3         | 6.2               | Insoluble.                 | Cracks and fuses.                    |
| Minium.       | Pb <sub>3</sub> O <sub>4</sub>  | Red and yellow.                | 2—3       | 4.6               | Soluble.                   | Lead Globules.                       |
| Pyromorphite. | Pb <sub>3</sub> P <sub>2</sub> O <sub>8</sub> + $\frac{1}{2}$ PbCl <sub>2</sub> | Green, yellow, brown, blue.    | 3.5—4     | 6—7               | Soluble.                   | Fusible, lead coat.                  |

## ANTIMONY.

|           |  |                        |       |         |                          |   |
|-----------|--|------------------------|-------|---------|--------------------------|---|
| Native.   | Sb   | Tin-white.             | 3—3.5 | 6.7     | Hot muriatic.            | Fuses, white fumes.                     |
| Stibnite. | Sb <sub>2</sub> S <sub>3</sub>                                   | Lead-gray to blackish. | 2     | 4.5—4.6 | Soluble.                 | Fuses easily and gives off white fumes. |
| Oxides.   | Sb <sub>2</sub> O <sub>3</sub><br>Sb <sub>2</sub> O <sub>5</sub> | Variable.              | 2—3   | 5—5.5   | Muriatic and aqua regia. | Fuses.                                  |

## SILVER.

|  |               |           |           |           |                             |  |
|--|---------------|-----------|-----------|-----------|-----------------------------|--|
| Native silver and alloys. Compounds with S. As. Sb. Cl. Br. I. Te. Se etc. | See page 249. | Variable. | Variable. | Variable. | Nitric acid and aqua regia. | Give, when fused on charcoal with soda and test lead, and the button cupelled, a bead of silver. |
|--|---------------|-----------|-----------|-----------|-----------------------------|--|

## GOLD.

|  |               |                   |       |       |             |   |
|--|---------------|-------------------|-------|-------|-------------|---|
| Native and alloys. Compounds with Te and Se. | See page 250. | Generally yellow. | 2.5—3 | 15—19 | Aqua regia. | Fusible to a bead, better with borax glass. |
|--|---------------|-------------------|-------|-------|-------------|---|

## PLATINUM.

|                    |                      |                         |       |         |             |            |
|--------------------|----------------------|-------------------------|-------|---------|-------------|------------|
| Native and alloys. | Pb. Ir. Au. Pd. etc. | Whitish, blue and gray. | 4—4.5 | 16—19.3 | Aqua regia. | Infusible. |
|--------------------|----------------------|-------------------------|-------|---------|-------------|------------|

## ZINC.

| ORES.        | COMPOSITION.  | COLOR.                    | HARDNESS. | SPECIFIC GRAVITY. | SOLUBILITY. SOLVENTS.                   | ON CHARCOAL, BEFORE THE BLOWPIPE.        |
|--------------|---|---------------------------|-----------|-------------------|---|--|
| Blende.      | ZnS   | Variable.                 | 3.5—4     | 3.9—4             | Nitric, and gives off H <sub>2</sub> S. | Infusible. Reaction for zinc.            |
| Smithsonite. | ZnCO <sub>3</sub>                                   | White to brown.           | 5         | 4—4.4             | Nitric and other acids. Effervesces.    | Infusible. Reaction for zinc.            |
| Calamine.    | Zn <sub>2</sub> SiO <sub>4</sub> + H <sub>2</sub> O | Variable.                 | 5         | 3.3—3.5           | Gelatinizes.                            | Fuses with difficulty. Reaction for zinc |
| Willemite.   | ZnSiO <sub>3</sub>                                  | Green, red, yellow, gray. | 5.5       | 3.8—4             | Muriatic acid.                          | Fusible with difficulty. Zinc coat.      |
| Zincite.     | ZnO   | Red.                      | 4—4.5     | 5.4—5.8           | All acids.                              | Infusible. Reaction for zinc.            |

## MERCURY.

|           |                                 |                 |       |      |              |                                       |
|-----------|---------------------------------|-----------------|-------|------|--------------|---------------------------------------|
| Mercury.  | Hg                              | Tin-white.      | —1    | 13.5 | Nitric acid. | Volatile.                             |
| Cinnabar. | HgS                             | Red.            | 2—2.5 | 8.1  | Aqua regia.  | Volatile, gives fumes SO <sub>2</sub> |
| Calomel.  | Hg <sub>2</sub> Cl <sub>2</sub> | White to brown. | 1—2   | 6.4  | Aqua regia.  | Volatile. White coat.                 |

## BISMUTH.

|           |                             |                         |                        |                      |                            |                                      |
|-----------|-----------------------------|-------------------------|------------------------|----------------------|----------------------------|--------------------------------------|
| Native.   | Bi                          | Reddish to white.       | When pure metal. 2—2.5 | When pure metal. 9.7 | Nitric and muriatic acids. | Easily fusible.                      |
| Oxide.    | Sometimes with Cu. Pb. etc. | Variable when combined. | Variable.              | Variable.            | “                          | Volatile, giving orange-yellow coat. |
| Sulphide. |                             |                         |                        |                      |                            |                                      |
| Arsenide. |                             |                         |                        |                      |                            |                                      |

## TIN.

|              |   |                      |     |         |             |  |
|--------------|---|----------------------|-----|---------|-------------|--|
| Cassiterite. | SnO <sub>2</sub>                              | Variable.            | 6—7 | 6.8—7.1 | Insoluble.  | Infusible. Reaction for tin.             |
| Stannite.    | Cu <sub>2</sub> S }<br>FeS } SnS <sub>2</sub> | Steel gray to black. | 4   | 4.5     | Aqua regia. | Gives fumes of sulphurous acid and fuses |

## COPPER.

|   |              |           |       |                         |              |   |
|---|--------------|-----------|-------|-------------------------|--------------|---|
| Native and alloys, oxidized ores and compounds, with S, As, Sb., etc. | See page 82. | Variable. | 2—4.5 | Native, 8.9; ores, 4—6. | Nitric acid. | Fusible or reducible to metal, reaction for copper. |
|---|--------------|-----------|-------|-------------------------|--------------|---|



## IRON.

| ORES.                   | COMPOSITION.   | COLOR.                         | HARDNESS. | SPECIFIC GRAVITY. | SOLUBILITY. SOLVENTS.                    | ON CHARCOAL, BEFORE THE BLOWPIPE.                    |
|-------------------------|--|--------------------------------|-----------|-------------------|--|--|
| Magnetite.              | $\text{Fe}_3\text{O}_4$  | Black, magnetic.               | 5.5—6.5   | 4.9—5.3           | Muriatic and aqua regia.                 | Infusible.   |
| Hematite.               | $\text{Fe}_2\text{O}_3$  | Red to black.                  | 5—6.5     | 4.9—5.3           | Muriatic and aqua regia.                 | Infusible, becomes magnetic.                         |
| Limonite.               | $2\text{Fe}_2\text{O}_3 + 3\text{H}_2\text{O}$                                       | Dark-brown.                    | 5—5.5     | 3.6—4             | Muriatic, warm.                          | Infusible, becomes magnetic.                         |
| Siderite.               | $\text{FeCO}_3$  | Variable.                      | 3.5—4     | 3.7—3.9           | Soluble with effervescence in hot acids. | Infusible, becomes magnetic.                         |
| Menaccanite (Ilmenite). | $\text{FeTiO}_3 + n\text{Fe}_2\text{O}_3$  | Iron-black, slightly magnetic. | 5—6       | 4.5—5             | Aqua regia.                              | Infusible.   |
| Franklinite.            | $(\text{FeO}, \text{ZnO}, \text{MnO})(\text{Fe}_2\text{O}_3, \text{Mn}_2\text{O}_3)$ | Black.                         | 6—6.5     | 5.1               | Warm muriatic.                           | Infusible, reaction for zinc.                        |
| Pyrite.                 | $\text{FeS}_2$   | Yellow.                        | 6—6.5     | 4.9—5.3           | Nitric acid.                             | Gives off $\text{SO}_2$ and becomes magnetic. Fuses. |
| Pyrrhotite.             | $\text{Fe}_7\text{S}_8$  | Yellow.                        | 3.5—4.5   | 4.5               | Nitric acid.                             | Magnetic Globules.                                   |
| Chromite.               | $\text{FeCr}_2\text{O}_4$  | Black.                         | 5.5       | 4.8—4.6           | Insoluble.                               | Infusible.   |

## MANGANESE.

|                |               |                      |     |     |                               |                                    |
|----------------|---------------|----------------------|-----|-----|-------------------------------|------------------------------------|
| Oxidized ores. | See page 171. | Dark-brown to black. | 1—6 | 3—5 | Conc. muriatic; gives off Cl. | Infusible. Reaction for manganese. |
|----------------|---------------|----------------------|-----|-----|-------------------------------|------------------------------------|

## NICKEL AND COBALT.

|            |  |                         |       |         |                             |   |
|------------|--|-------------------------|-------|---------|-----------------------------|---|
| Niccolite. | $\text{NiAs}$                                  | Copper-red.             | 5—5.5 | 7.3—7.6 | Aqua regia. Green solution. | Fuses with odor of As. Reaction for Ni.               |
| Millerite. | $\text{NiS}$                                   | Brass or bronze-yellow. | 3—3.5 | 5.3—5.6 | Aqua regia.                 | Fuses to a brittle magnetic globule. Reaction for Ni. |
| Smaltite.  | $(\text{Co}, \text{Fe}, \text{Ni})\text{As}_2$ | Tin-white to gray.      | 5.5—6 | 6.4—6.6 | Nitric acid. Pink sol.      | Gives off As, melts, and reaction for Co.             |
| Cobaltite. | $\text{CoS}_2 + \text{CoAs}_2$                 | Silver-white to gray.   | 5.5   | 6—6.3   | Nitric acid.                | Gives off As, and becomes magnetic.                   |
| Linnéite.  | $\text{Co}_3\text{S}_4$                        | Yellow.                 | 5.5   | 4.8—5   | Nitric acid.                | Fusible. Reaction for cobalt.                         |

CARBON.

| ORES.                                       | COMPOSITION.      | COLOR.              | HARDNESS. | SPECIFIC GRAVITY. | SOLUBILITY. SOLVENTS. | ON CHARCOAL, BEFORE THE BLOWPIPE.                      |
|---|-------------------|---------------------|-----------|-------------------|-----------------------|--|
| Diamond, Graphite, Coals, Lignite and Wood. | Pure to variable. | Colorless to black. | Variable. | Variable.         | Insoluble.            | Burn, leaving an ash, save in the case of the diamond. |

SULPHUR.

| Sulphur and Sulphides. | Variable. | Yellow, when pure. | Variable. | Variable. | When pure, in bi-sulphide of carbon. | Melt and burn, giving off SO <sub>2</sub> in most cases. |
|------------------------|-----------|--------------------|-----------|-----------|--------------------------------------|--|
|------------------------|-----------|--------------------|-----------|-----------|--------------------------------------|--|

SILVER—ORES AND MINERALS.

| Mineral.                    | Composition.  | Per Cent. of Silver, when pure. |
|-----------------------------|---|---------------------------------|
| Naumannite..                | Ag <sub>2</sub> Se .....  | 73.2                            |
| Eucairite.....              | Cu <sub>2</sub> Se + Ag <sub>2</sub> Se .....   | 43.1                            |
| Hessite.....                | Ag <sub>2</sub> Te .....  | 63.3                            |
| Petzite.....                | (Au, Ag) <sub>2</sub> Te .....  | (Variable,) 42.0                |
| Sylvanite ....              | (Au, Ag)Te <sub>3</sub> .....   | 10 to 15                        |
| Silver Glance.              | Ag <sub>2</sub> S (Argentite) .....   | 87.1                            |
| Stromeyerite..              | Ag <sub>2</sub> S + Cu <sub>2</sub> S .....   | 53.1                            |
| Sternbergite...             | AgFe <sub>2</sub> S <sub>3</sub> .....  | 34.2                            |
| Miargyrite....              | Ag <sub>2</sub> S + Sb <sub>2</sub> S <sub>3</sub> .....                                  | 36.9                            |
| Pyrargyrite...              | 3Ag <sub>2</sub> S + Sb <sub>2</sub> S <sub>3</sub> .....                                 | 59.9                            |
| Proustite.....              | 3Ag <sub>2</sub> S + As <sub>2</sub> S <sub>3</sub> .....                                 | 65.4                            |
| Stephanite....              | 5Ag <sub>2</sub> S + Sb <sub>2</sub> S <sub>3</sub> .....                                 | 68.5                            |
| Xanthoconite.               | 3Ag <sub>2</sub> S, As <sub>2</sub> S <sub>3</sub> .....                                  | 61.4                            |
| Polybasite....              | 9(Ag <sub>2</sub> Cu)S + (Sb, As) <sub>2</sub> S <sub>3</sub> .....                       | 75.6                            |
| Tetrahedrite {<br>(Fahlerz) | { (Cu, Ag) <sub>2</sub> S + (Sb, As, Bi) <sub>2</sub> S <sub>3</sub> +<br>(Fe, Zn, Hg)S } | ... Variable                    |
| Fireblende....              | Ag <sub>3</sub> SbS <sub>3</sub> (Pyrostilpnite) .....                                    | 59.9                            |
| Freieslebenite.             | (PbAg <sub>2</sub> ) <sub>5</sub> Sb <sub>4</sub> S <sub>11</sub> .....                   | 24.5                            |
| Cerargyrite...              | AgCl .....  | 75.33                           |
| Bromyrite....               | AgBr .....  | 57.4                            |



## GOLD—ALLOYS.

Native Gold.....AuAg—Au 65 to 99  
 Palladium Gold (Porpezite).....AuPd—Au 85.98—Ag 4.17  
 Rhodium Gold.....AuRd—Au 59 to 66  
 Gold Amalgam.....(Au, Ag)<sub>4</sub>Hg<sub>5</sub>—Au 38.39—Ag 5  
 Artificial Alloys, Gold Coin, Jewelry, etc.

The preceding list does not include all of the *rare* minerals of gold and silver; for these the reader is referred to Dana's System of Mineralogy.

WEIGHT OF ONE CUBIC FOOT, AND VOLUME OF ONE TON,  
 OF SOME IMPORTANT MINERALS.

1 cubic foot of water = 62.4 lbs.

| MINERAL.                               | WEIGHT<br>OF CUBIC<br>FOOT. | CUBIC<br>FEET IN<br>ONE TON. | MINERAL.                               | WEIGHT<br>OF CUBIC<br>FOOT. | CUBIC<br>FEET IN<br>ONE TON. |
|--|-----------------------------|------------------------------|--|-----------------------------|------------------------------|
| Quartz.....                            | 164 lbs.                    | 12.19                        | Chalcopyrite.....<br>(Copper Pyrites.) | 262 lbs.                    | 7.63                         |
| Argentite.....<br>(Silver Glance.)     | 454 "                       | 4.40                         | Tetrahedrite.....<br>(Gray Copper.)    | 300 "                       | 6.66                         |
| Pyragryrite.....<br>(Ruby Silver.)     | 363 "                       | 5.51                         | Galenite.....<br>(Galen.)              | 468 "                       | 4.27                         |
| Proustite.....<br>(Light Ruby Silver.) | 350 "                       | 5.71                         | Sphalerite.....<br>(Blende.)           | 250 "                       | 8.00                         |
| Stephanite.....<br>(Black Silver.)     | 390 "                       | 5.13                         | Pyrite.....<br>(Iron Pyrites.)         | 318 "                       | 6.29                         |
| Cerargyrite.....<br>(Horn Silver.)     | 328 "                       | 6.10                         | Limestone.....<br>(Calcite.)           | 170 "                       | 11.76                        |
| Stibnite.....<br>(Antimony Glance.)    | 285 "                       | 7.02                         | Kaolin... ..<br>(Clay.)                | 162 "                       | 12.34                        |
| Cinnabar.....                          | 505 "                       | 3.96                         |  |                             |                              |

NOTE.—The values given above are for the pure minerals, and can be obtained by multiplying the weight of one cubic foot of water by the specific gravity of the mineral; the product = the weight of a cubic foot of the mineral or ore; 2000 divided by this = volume of one ton.

## COINS OF THE UNITED STATES.

(By Act of Congress, February 12, 1873.)

## GOLD COINS.

| Denomination.         | Weight.     | Fineness. |
|-----------------------|-------------|-----------|
| Dollar, unit of value | 25.8 grains | 900       |
| Quarter Eagle, \$2.50 | 64.5 "      | "         |
| Three Dollars. ....   | 77.4 "      | "         |
| Half Eagle, \$5. .... | 129.0 "     | "         |
| Eagle, \$10. ....     | 258.0 "     | "         |
| Double Eagle, \$20 .. | 516.0 "     | "         |

## SILVER COINS.

|                       |              |     |
|-----------------------|--------------|-----|
| Dollar. ....          | 412.5 grains | 900 |
| Half Dollar, 50c. ... | 192.0 "      | "   |
| Quarter " 25c. ...    | 96.0 "       | "   |
| Dime, 10c. ....       | 38.4 "       | "   |

## MINOR COINS.

|                       |              |   |
|-----------------------|--------------|---|
| Five-cent piece. .... | 77.16 grains | Cu 75%, Ni 25%                          |
| Three " " ....        | 32.0 "       | " " " "                                 |
| One " " ....          | 48.0 "       | Cu 95%, Sn and Zn<br>5%, (3% Sn, 2% Zn) |

## FOREIGN COINS.

|                  | Gold.     | Fineness. | Silver.         | Fineness. |
|------------------|-----------|-----------|-----------------|-----------|
| Austria. ....    | Gulden    | 900       | Gulden          | 900       |
| France. ....     | Franc     | 900       | Franc           | 900       |
| German Empire    | Mark      | 900       | Mark            | 900       |
| Great Britain. . | Sovereign | 916.667   | Crown, Shilling | 925       |
| Russia. ....     | Dukat     | 916.667   | Rouble          | 868.056   |

## MEASURES OF WEIGHT AND VOLUME.

## AVOIRDUPOIS WEIGHT.

Used for weighing base metals, as lead, antimony, tin, etc.

|                       |                        |            |
|-----------------------|------------------------|------------|
| 15 Drams (dr.) .....  | make 1 ounce .....     | marked oz. |
| 16 Ounces .....       | make 1 pound .....     | " lb.      |
| 25 Pounds .....       | make 1 quarter .....   | " qr.      |
| 4 Quarters .....      | make 1 hundred-weight. | " cwt.     |
| 20 Hundred-weight ... | make 1 ton .....       | " t.       |

## TROY WEIGHT.

Used for weighing precious metals, as gold, silver, etc.

|                       |                        |        |
|-----------------------|------------------------|--------|
| 24 Grains (gr.) ..... | make 1 pennyweight ... | " dwt. |
| 20 Pennyweights ..... | make 1 ounce .....     | " oz.  |
| 12 Ounces .....       | make 1 pound .....     | " lb.  |

FRENCH OR DECIMAL WEIGHTS.

Used in weighing all metals.

|                     |                       |                 |
|---------------------|-----------------------|-----------------|
| 10 Milligrams (mg.) | .....make 1 centigram | .....marked cg. |
| 10 Centigrams       | .....make 1 decigram  | ..... " dcg.    |
| 10 Decigrams        | .....make 1 gram      | ..... " gm.     |
| 10 Grams            | .....make 1 decagram  | ..... " dkg.    |
| 10 Decagrams        | .....make 1 hectogram | ..... " hg.     |
| 10 Hectograms       | .....make 1 kilogram  | ..... " kg.     |
| 10 Kilograms        | .....make 1 myriagram | ..... " myrg.   |

The unit of the system is the gram = 15.438395 Troy grains, or the weight of 1 c.c. distilled water at 4° C.

ASSAY WEIGHTS.

|              |      |   |   |
|--------------|------|---|---|
| Multiples    | {    | 4 Assay Tons = 116.66666 grams.         |   |
|              |      | 2 Assay Tons = 58.33333                 | " |
| Unit         | .... | The Assay Ton (marked A. T.) = 29.16666 | " |
| Subdivisions | {    | $\frac{1}{8}$ Assay Ton = 9.7222        | " |
|              |      | $\frac{1}{4}$ " " = 4.8611              | " |
|              |      | $\frac{1}{10}$ " " = 2.9166             | " |
|              |      | $\frac{1}{20}$ " " = 1.4583             | " |

LIQUID MEASURE—UNITED STATES.

|               |                    |                 |
|---------------|--------------------|-----------------|
| 4 Gills (gi.) | .....make 1 pint   | .....marked pt. |
| 2 Pints       | .....make 1 quart  | ..... " qt.     |
| 4 Quarts      | .....make 1 gallon | ..... " gal.    |

LIQUID MEASURE—FRENCH.

|                      |                        |                 |
|----------------------|------------------------|-----------------|
| 10 Milliliters (ml.) | .....make 1 centiliter | .....marked cl. |
| 10 Centiliters       | .....make 1 deciliter  | ..... " dcl.    |
| 10 Deciliters        | .....make 1 liter      | ..... " l.      |
| 10 Liters            | .....make 1 decaliter  | ..... " dkl.    |
| 10 Decaliters        | .....make 1 hectoliter | ..... " hl.     |
| 10 Hectoliters       | .....make 1 kiloliter  | ..... " kl.     |

The unit is 1 liter = 61.0295688 cubic inches, or 2.1134 pints (1.760773 imperial pints.)

## CUBIC MEASURE.—ENGLISH.

|                            |                    |                |
|----------------------------|--------------------|----------------|
| 1728 Cubic inches (c. in.) | make 1 cubic foot, | marked cu. ft. |
| 27 Cubic feet.....         | make 1 cubic yard  | “ c. yd.       |
| 16 Cubic feet.....         | make 1 cord foot.. | “ c. ft.       |
| 8 Cord ft. or 128 cu. ft.  | make 1 cord.....   | “ c.           |

## CUBIC MEASURE.—FRENCH.

1000 Cubic centimeters (c. c.) make one cubic decimeter or litre, marked l.

1000 Cubic decimeters make 1 cubic meter or kiloliter, marked kl. or cu. m.

Otherwise the cubic measure is the same as liquid.

## COMPARISON OF UNITS.

|                           |   |
|---------------------------|---|
| 1 Meter.....              | =39.37079 inches.                               |
| 1 Are.....                | =(393.7079) <sup>2</sup> =155005.91 sq. inches. |
| 1 Liter.....              | =(3.937079) <sup>3</sup> =61.027 cubic inches.  |
| 1 Pound Avoirdupois...    | =7000 grains Troy.                              |
| 1 Pint, U. S.....         | =28.875 cubic inches.                           |
| 1 English ton (2240 lbs.) | =15,680,000 grains.                             |
| 1 Short ton (2000 lbs.).. | =14,000,000 grains.                             |

## SPECIFIC GRAVITY.

The specific gravity of a body is the weight of that body as compared with the weight of an equal volume of another body, assumed as a standard.

The standard for solids and liquids is distilled water; for gases and vapors, dry air and sometimes hydrogen.

All determinations must be made at known temperatures ; this for solids and liquids is 60° F. or 15.5° C.

Gases and vapors may be observed at any known temperature, and the volume reduced by calculation to what it would be at 0° C.

Formulæ for the determination of the specific gravity of solids and liquids :

a. Solids.

1. The substance is heavier than water, and insoluble in it. Weigh it in air and then in water :

Let the weight of the substance in air =  $W$ .

Let the weight of the substance in water =  $W'$ .

The specific gravity =  $\frac{W}{W - W'}$ .

2. The substance is heavier than water and insoluble in it :

Fill a flask to any fixed mark on the neck, with water, and weigh ; the weight =  $W'$ .  $W$  = weight of the substance. Place it in the flask and reduce the water to the same level, then weigh the flask, plus substance and water left. Let this weight =  $W''$ .

The specific gravity =  $\frac{W}{(W + W') - W''}$ .

3. The substance is heavier than water, but in fragments, and is insoluble. Use the same method as in 2.

4. The substance is heavier than water, but soluble in it: Weigh in some liquid of known specific gravity in which it is insoluble, in place of water. Calculate as follows :

Weight of substance in air =  $W$ .

Weight of substance in liquid =  $W'$ .

Specific gravity of liquid =  $S$ .

Specific gravity of water = 1.



The liquid displaced  $= W - W' = X$ .

Then S is to 1 as X is to  $W''$  = the water that would have been displaced.

The specific gravity  $= \frac{W}{W''}$

5. The substance is insoluble, but lighter than water :

Weight of the substance in air  $= W$ .

Then weigh in water with a piece of lead attached to sink it. Let this  $= W'$ .

Weight of the lead alone in water  $= W''$

The specific gravity  $= \frac{W}{W - (W' + W'')}$

6. The substance is soluble, but lighter than water.

Use the process by the flask as in 2, but substitute benzine or turpentine for water.

Weight in air  $= W$  ; in liquid  $= W'$ . Specific gravity of liquid  $= S$ . Water  $= 1$ .  $W - W' = W'' =$  liquid displaced. S is to 1 as  $W''$  is to  $X =$  the water that would have been displaced. Specific gravity  $= \frac{W}{X}$

b. Liquids.

Three methods may be employed.

1. By the specific gravity bottle. This is a thin glass flask with a hollow stopper, so as to allow the insertion of a thermometer.

Weight of the flask  $= W$ .

“ “ “ “ filled with water  $= W'$ .

“ “ “ “ “ the liquid  $= W''$ .

Specific gravity  $= \frac{W'' - W}{W' - W}$

2. By weighing some body first in water and then in the liquid.

The body weighed in air  $= W$ .

“ “ “ “ water  $= W'$ .

“ “ “ “ liquid  $= W''$ .

Specific gravity  $= \frac{W'' - W}{W' - W}$

3. By means of the hydrometer, which is an instrument that, placed in a liquid, shows its specific gravity by direct inspection. Its action depends upon the simple principle that a floating body displaces its own weight of liquid. Hydrometers vary in construction according to the purposes for which they are to be used, but are generally made of light glass tubes with bulbs, blown in a single piece; the weight desired being given by means of small shot or mercury placed in the bulb at the lower end, which is afterwards carefully sealed.

The graduation may be made according to the true scale of specific gravities or arbitrarily; the first is, of course, most desirable and generally employed. For commercial purposes the Baumé scale is often used; it is arbitrary, and is determined by marking the point to which the instrument sinks in pure water "0," and the point to which it sinks in a solution of 15 parts of salt in 85 of water at 12.5°C, "15," the interval being divided into 15 equal parts. For liquids lighter than water a 10% solution of salt gives the zero point while distilled water gives the 10 point. (See tables.)

Twaddle's hydrometer gives the specific gravity by a simple calculation: on this 200° represent the densities between 1 and 2 and 1° represents a difference of specific gravity of 0.005; hence the sp. gr =  $1 + \frac{n^\circ (\text{Twaddle})}{200}$ .

4. The Westphal balance is a very accurate apparatus for the rapid determination of the specific gravity of liquids. It consists of a graduated brass beam set on knife-edges, from one end of which is suspended a float. The liquid to be tested is placed in a glass cylinder about 6 inches high, the float introduced, and weights placed on the beam until it is level; the specific gravity is then read from the weights used. The weights are arranged so that each size gives the figure for a decimal place.

# TABLE OF COMPARISON—DEGREES OF THE BAUMÉ HYDROMETER AND SPECIFIC GRAVITY.\*

TABLE FOR LIQUIDS HEAVIER THAN WATER.

| DEGREES<br>BAUMÉ. | SPECIFIC<br>GRAVITY. | DEGREES<br>BAUMÉ. | SPECIFIC<br>GRAVITY. | DEGREES<br>BAUMÉ. | SPECIFIC<br>GRAVITY. |
|-------------------|----------------------|-------------------|----------------------|-------------------|----------------------|
| 0                 | 1.000                | 26                | 1.206                | 52                | 1.520                |
| 1                 | 1.007                | 27                | 1.216                | 53                | 1.535                |
| 2                 | 1.013                | 28                | 1.226                | 54                | 1.551                |
| 3                 | 1.020                | 29                | 1.236                | 55                | 1.567                |
| 4                 | 1.027                | 30                | 1.246                | 56                | 1.583                |
| 5                 | 1.034                | 31                | 1.256                | 57                | 1.600                |
| 6                 | 1.041                | 32                | 1.267                | 58                | 1.617                |
| 7                 | 1.048                | 33                | 1.277                | 59                | 1.634                |
| 8                 | 1.056                | 34                | 1.288                | 60                | 1.652                |
| 9                 | 1.063                | 35                | 1.299                | 61                | 1.670                |
| 10                | 1.070                | 36                | 1.310                | 62                | 1.689                |
| 11                | 1.078                | 37                | 1.322                | 63                | 1.708                |
| 12                | 1.086                | 38                | 1.333                | 64                | 1.727                |
| 13                | 1.094                | 39                | 1.345                | 65                | 1.747                |
| 14                | 1.101                | 40                | 1.357                | 66                | 1.767                |
| 15                | 1.109                | 41                | 1.369                | 67                | 1.788                |
| 16                | 1.118                | 42                | 1.382                | 68                | 1.809                |
| 17                | 1.126                | 43                | 1.395                | 69                | 1.831                |
| 18                | 1.134                | 44                | 1.407                | 70                | 1.854                |
| 19                | 1.143                | 45                | 1.421                | 71                | 1.877                |
| 20                | 1.152                | 46                | 1.434                | 72                | 1.900                |
| 21                | 1.160                | 47                | 1.448                | 73                | 1.924                |
| 22                | 1.169                | 48                | 1.462                | 74                | 1.949                |
| 23                | 1.178                | 49                | 1.476                | 75                | 1.974                |
| 24                | 1.188                | 50                | 1.490                | 76                | 2.000                |
| 25                | 1.197                | 51                | 1.505                |                   |                      |

TABLE FOR LIQUIDS LIGHTER THAN WATER.

| DEGREES<br>BAUMÉ. | SPECIFIC<br>GRAVITY. | DEGREES<br>BAUMÉ. | SPECIFIC<br>GRAVITY. | DEGREES<br>BAUMÉ. | SPECIFIC<br>GRAVITY. |
|-------------------|----------------------|-------------------|----------------------|-------------------|----------------------|
| 10                | 1.000                | 27                | .896                 | 44                | .811                 |
| 11                | .993                 | 28                | .890                 | 45                | .807                 |
| 12                | .986                 | 29                | .885                 | 46                | .802                 |
| 13                | .980                 | 30                | .880                 | 47                | .798                 |
| 14                | .973                 | 31                | .874                 | 48                | .794                 |
| 15                | .967                 | 32                | .869                 | 49                | .789                 |
| 16                | .960                 | 33                | .864                 | 50                | .785                 |
| 17                | .954                 | 34                | .859                 | 51                | .781                 |
| 18                | .948                 | 35                | .854                 | 52                | .777                 |
| 19                | .942                 | 36                | .849                 | 53                | .773                 |
| 20                | .936                 | 37                | .844                 | 54                | .768                 |
| 21                | .930                 | 38                | .839                 | 55                | .764                 |
| 22                | .924                 | 39                | .834                 | 56                | .760                 |
| 23                | .918                 | 40                | .830                 | 57                | .757                 |
| 24                | .913                 | 41                | .825                 | 58                | .753                 |
| 25                | .907                 | 42                | .820                 | 59                | .749                 |
| 26                | .901                 | 43                | .816                 | 60                | .745                 |

\* Crooke's Select Methods, p. 693.

## THERMOMETERS.

Three scales are now in general use. These are:

1. Centigrade—C. Water freezes at 0°, boils at 100°.
2. Fahrenheit—F. “ “ 32°, “ 212°.
3. Réaumur—R. “ “ 0°, “ 80°.

$$\left. \begin{array}{l} \text{To CONVERT—F. to C. } \frac{5(\text{F.}^\circ - 32^\circ)}{9} = \text{C.}^\circ \\ \text{C. to F. } \frac{9\text{C.}^\circ}{5} + 32^\circ = \text{F.}^\circ \\ \text{R. to F. } \frac{9\text{R.}^\circ}{4} + 32^\circ = \text{F.}^\circ \end{array} \right\} \text{Formulæ.}$$

## TABLE OF VALUES FOR GRAIN WEIGHTS.

| If 240 grains of ore give of fine metal—Thousands of 10 grains..... | One Ton of Ore will yield in Troy ounces..... | Coin Value per Ton—Gold. | If 240 grains of Ore give of fine Metal—Thousands of 10 grains..... | One Ton of Ore will yield in Troy ounces..... | Coin Value per Ton—Gold. | If 240 grains of Ore give of fine Metal—Thousands of 10 grains..... | One Ton of Ore will yield in Troy ounces..... | Coin Value per Ton—Gold. |
|---|---|--------------------------|---|---|--------------------------|---|---|--------------------------|
|   |   |                          |   |   |                          |   |   |                          |
| .001  | 1.21  | \$ 25.11                 | 7   | 44.95   | \$ 929.19                | 3   | 88.69   | \$ 1,883.87              |
| 2   | 2.43  | 50.23                    | 8   | 46.17   | 954.42                   | 4   | 89.91   | 1,858.59                 |
| 3   | 3.64  | 75.24                    | 9   | 47.38   | 979.43                   | 5   | 91.12   | 1,833.60                 |
| 4   | 4.86  | 100.46                   | .040  | 48.60   | 1,004.65                 | 6   | 92.34   | 1,808.82                 |
| 5   | 6.07  | 125.48                   | 1   | 49.81   | 1,029.66                 | 7   | 93.55   | 1,783.88                 |
| 6   | 7.29  | 150.70                   | 2   | 51.03   | 1,054.88                 | 8   | 94.77   | 1,759.06                 |
| 7   | 8.50  | 175.71                   | 3   | 52.24   | 1,079.89                 | 9   | 95.98   | 1,734.06                 |
| 8   | 9.72  | 200.93                   | 4   | 53.46   | 1,105.11                 | .060  | 97.20   | 2,009.28                 |
| 9   | 10.93   | 225.94                   | 5   | 54.67   | 1,130.12                 | 1   | 98.41   | 2,034.29                 |
| .010  | 12.15   | 251.16                   | 6   | 55.89   | 1,155.34                 | 2   | 99.63   | 2,059.51                 |
| 1   | 13.39   | 276.17                   | 7   | 57.10   | 1,180.35                 | 3   | 100.84  | 2,084.52                 |
| 2   | 14.58   | 301.39                   | 8   | 58.32   | 1,205.57                 | 4   | 102.06  | 2,109.74                 |
| 3   | 15.79   | 326.41                   | 9   | 59.53   | 1,230.58                 | 5   | 103.27  | 2,134.75                 |
| 4   | 17.01   | 351.63                   | .050  | 60.75   | 1,255.80                 | 6   | 104.49  | 2,159.97                 |
| 5   | 18.22   | 376.64                   | 1   | 61.96   | 1,280.81                 | 7   | 105.70  | 2,184.98                 |
| 6   | 19.43   | 401.65                   | 2   | 63.18   | 1,306.03                 | 8   | 106.92  | 2,210.20                 |
| 7   | 20.65   | 426.87                   | 3   | 64.39   | 1,331.04                 | 9   | 108.13  | 2,235.21                 |
| 8   | 21.86   | 451.88                   | 4   | 65.61   | 1,356.26                 | .090  | 109.35  | 2,260.43                 |
| 9   | 23.08   | 477.10                   | 5   | 66.82   | 1,381.27                 | 1   | 110.56  | 2,285.44                 |
| .020  | 24.30   | 502.32                   | 6   | 68.04   | 1,406.49                 | 2   | 111.78  | 2,310.66                 |
| 1   | 25.51   | 527.34                   | 7   | 69.25   | 1,431.50                 | 3   | 112.99  | 2,335.67                 |
| 2   | 26.73   | 552.56                   | 8   | 70.47   | 1,456.72                 | 4   | 114.21  | 2,360.89                 |
| 3   | 27.94   | 577.57                   | 9   | 71.68   | 1,481.73                 | 5   | 115.42  | 2,385.90                 |
| 4   | 29.16   | 602.79                   | .060  | 72.90   | 1,506.95                 | 6   | 116.64  | 2,411.12                 |
| 5   | 30.37   | 627.80                   | 1   | 74.11   | 1,531.96                 | 7   | 117.85  | 2,436.13                 |
| 6   | 31.59   | 653.02                   | 2   | 75.33   | 1,557.18                 | 8   | 119.07  | 2,461.35                 |
| 7   | 32.80   | 678.03                   | 3   | 76.54   | 1,582.19                 | 9   | 120.28  | 2,486.36                 |
| 8   | 34.02   | 703.25                   | 4   | 77.76   | 1,607.41                 | .100  | 121.50  | 2,511.62                 |
| 9   | 35.23   | 728.26                   | 5   | 78.97   | 1,632.42                 | .200  | 243.00  | 5,023.25                 |
| .080  | 36.45   | 753.48                   | 6   | 80.19   | 1,657.44                 | .300  | 364.50  | 7,131.77                 |
| 1   | 37.66   | 778.50                   | 7   | 81.40   | 1,682.65                 | .400  | 486.00  | 10,046.50                |
| 2   | 38.88   | 803.72                   | 8   | 82.62   | 1,707.87                 | .500  | 607.50  | 15,558.12                |
| 3   | 40.09   | 828.73                   | 9   | 83.83   | 1,732.88                 | .600  | 729.00  | 14,263.55                |
| 4   | 41.31   | 853.95                   | .070  | 85.05   | 1,758.13                 | .700  | 850.50  | 17,581.37                |
| 5   | 42.52   | 879.06                   | 1   | 86.26   | 1,783.14                 | .800  | 972.00  | 20,093.00                |
| 6   | 43.74   | 904.18                   | 2   | 87.48   | 1,808.36                 | .900  | 1,093.50                                      | 22,604.61                |
|   |   |                          |   |   |                          | 1000  | 1,215.00                                      | 25,116.25                |

## MULTIPLICATION TABLE FOR GOLD.

|                          |                           |                           |
|--------------------------|---------------------------|---------------------------|
| $20.67 \times 1 = 20.67$ | $20.67 \times 4 = 82.68$  | $20.67 \times 7 = 144.69$ |
| $20.67 \times 2 = 41.34$ | $20.67 \times 5 = 103.35$ | $20.67 \times 8 = 165.36$ |
| $20.67 \times 3 = 62.01$ | $20.67 \times 6 = 124.02$ | $20.67 \times 9 = 186.03$ |

EXAMPLE.—Suppose an ore gave on assay  
218.6 oz., then.....

|                                       |
|---------------------------------------|
| \$4134.                               |
| 206.7                                 |
| 165.36                                |
| 12.402 added will give                |
| <hr/> \$4518.462 per ton of 2000 lbs. |

## GENERAL STYLE OF REPORT.

(Certificate.)

New York, \_\_\_\_\_ 18

DEAR SIR:

The sample of \_\_\_\_\_

From \_\_\_\_\_

Marked \_\_\_\_\_

\* \_\_\_\_\_ for examination, contains \_\_\_\_\_

Very respectfully,

To \_\_\_\_\_

\* Fill in *Sampled* or *Submitted*.

## QUANTITATIVE REPORT.

(For Reference.)

New York, \_\_\_\_\_ 18

Report of

Analysis of

Determination of

Weight taken

Method of Analysis.

| Precipitates. | Actual<br>Weights. | Constituents. | Calculated<br>Weights. | Percentages. |
|---------------|--------------------|---------------|------------------------|--------------|
|---------------|--------------------|---------------|------------------------|--------------|

Special Remarks

[illegible]

**Dated, Signed,**

**This form of report will answer for all base metals, except iron.**

## IRON—CRUCIBLE ASSAY.

Ore—Marked

Mineral Character

Composition

Alumina per cent.

Silica “

Lime “

Charge—Ore

Silica

Lime

Glass

Kaolin

Fluorspar

No. 1.

Gms.

No. 2.

Gms.

“

“

“

“

“

“

“

“

“

“

In fire—

Slag, Color

Appearance

Button, Wt.

Character

Hours.

Hours.

Gms.

Gms.

Remarks

## REPORT.

Assay

No. 1

per cent Iron.

No. 2

“ “ “

Average

“ “ “

Sample Averaged on

lbs. Ore

Dated

No.

Signed



**264 SILVER AND GOLD—CRUCIBLE ASSAY REPORT.**

**SILVER AND GOLD—CRUCIBLE ASSAY.**

**Ore—Marked**

**Mineral Character**

**Reducing Power**

**Gms. = Gms. Lead.**

|                                    | No. 1.       | No. 2.       |
|------------------------------------|--------------|--------------|
| <b>Charge—Ore</b>                  | <b>A. T.</b> | <b>A. T.</b> |
| Litharge                           | "            | "            |
| Carb. Potash or Soda               | "            | "            |
| Borax Glass                        | "            | "            |
| Silica                             | "            | "            |
| Charcoal or Argol                  | Gms.         | Gms.         |
| Nitre                              | "            | "            |
| Salt                               | "            | "            |
| <b>In fire—to fusion</b>           | <b>Mts.</b>  | <b>Mts.</b>  |
| after fusion                       | "            | "            |
| <b>Slag, Color</b>                 |              |              |
| Appearance                         |              |              |
| <b>Lead Button, Wt.</b>            | <b>Gms.</b>  | <b>Gms.</b>  |
| Character                          |              |              |
| <b>Scorification—Fluxes</b>        |              |              |
| Wt. after 1st.                     | Gms.         | Gms.         |
| " " 2nd.                           | "            | "            |
| <b>Cupellation—Silver and Gold</b> | <b>Mgs.</b>  | <b>Mgs.</b>  |
| Gold in Ore                        | "            | "            |
| Silver                             | "            | "            |
| Silver in Litharge                 | "            | "            |
| Silver in Ore                      | "            | "            |

**Remarks**

**REPORT.**

**Contained in 2,000 lbs. Ore**

| Assay   | Gold | Silver | Total | Gold | Silver | Total |
|---------|------|--------|-------|------|--------|-------|
| No. 1.  | oz.  | oz.    | oz.   | \$   | \$     | \$    |
| No. 2.  | oz.  | oz.    | oz.   | \$   | \$     | \$    |
| Average | oz.  | oz.    | oz.   | \$   | \$     | \$    |

**Sample Averaged on**

**Ore**

**Dated**

**No.**

**Signed**

## SILVER AND GOLD—SCORIFICATION ASSAY.

Ore—Marked

Mineral Character

|                             | No. 1. | No. 2. |
|-----------------------------|--------|--------|
| Charge—Ore                  | A. T.  | A. T.  |
| Test Lead                   | Gms.   | Gms.   |
| Borax Glass                 | "      | "      |
| Silica or Glass             | "      | "      |
| No. of Scorifiers           |        |        |
| Scorification               |        |        |
| Slag—Color                  |        |        |
| Appearance                  |        |        |
| Button—Character            |        |        |
| Weight                      |        |        |
| " after 2nd Scor.           | Gms.   | Gms.   |
| " " 3rd "                   | "      | "      |
| " " 4th "                   | "      | "      |
| " " 5th "                   | "      | "      |
| Cupellation—Silver and Gold | Mgs.   | Mgs.   |
| Gold in Ore                 | "      | "      |
| Silver                      | "      | "      |
| Silver in Test Lead         | "      | "      |
| Silver in Ore               | "      | "      |

Remarks

## REPORT.

Contained in 2,000 lbs. Ore.

| Assay   | Gold | Silver | Total | Gold | Silver | Total |
|---------|------|--------|-------|------|--------|-------|
| No. 1.  | oz.  | oz.    | oz.   | \$   | \$     | \$    |
| No. 2.  | oz.  | oz.    | oz.   | \$   | \$     | \$    |
| Average | oz.  | oz.    | oz.   | \$   | \$     | \$    |

Sample Averaged on

Ore

Dated

No.

Signed

## GOLD BULLION ASSAY.

Alloy—Marked

|                 | No. 1. | No. 2. |
|-----------------|--------|--------|
| Copper Assay    |        |        |
| Bullion         | Mgs.   | Mgs.   |
| Lead            | Gms.   | Gms.   |
| Gold and Silver | Mgs.   | Mgs.   |
| Base metal      | "      | "      |
| Assay proper    |        |        |
| Cupellation     |        |        |
| Bullion         | Mgs.   | Mgs.   |
| Silver          | "      | "      |
| Lead            | Gms.   | Gms.   |
| Copper          | "      | "      |
| Parting         |        |        |
| Cornet          | Mgs.   | Mgs.   |
| Surcharge       | "      | "      |
| Gold            | "      | "      |

Remarks

## REPORT.

| Assay   | Gold. | Silver. | Base Metal. |
|---------|-------|---------|-------------|
| No. 1.  | Fine  | Thds.   | Thds.       |
| No. 2.  | "     | "       | "           |
| Average | "     | "       | "           |

Dated

No.

Signed

## SILVER BULLION ASSAY.

## Alloy—Marked

## Cupellation

Bullion

Lead

Silver

Correction for Loss

Fineness

## Volumetric Assay

Bullion

Normal Salt solution

Decimal “ “

Total

Decimal Silver solution

Total Salt

Equivalent in Silver

Fineness

| No. 1.     | No. 2.    |
|------------|-----------|
| Mgs.       | Mgs.      |
| Gms.       | Gms.      |
| Mgs.       | Mgs.      |
| “          | “         |
|            |           |
| Gms.       | Gms.      |
| .100. c.c. | 100. c.c. |
| “          | “         |
| “          | “         |
| “          | “         |
| Gms.       | Gms.      |
| Thds.      | Thds.     |

## Remarks

## REPORT.

| Assay   | Silver. | Copper, &c |
|---------|---------|------------|
| No. 1.  | Fine.   | Thds.      |
| No. 2.  | “       | “          |
| Average | “       | “          |

Dated

No.

Signed

## SOLUBILITY OF COMMON ELEMENTS AND COMPOUNDS. (ALPHABETICALLY ARRANGED.)

*(Taken from Comey's Dictionary of Solubilities.)*

Aluminum (Al). Easily soluble in HCl, dilute or concentrated, hot or cold. Insoluble in cold dilute  $\text{H}_2\text{SO}_4$ , soluble in hot concentrated  $\text{H}_2\text{SO}_4$ , slightly soluble in  $\text{HNO}_3$ , soluble in caustic alkalis.

Aluminum hydroxide ( $\text{Al}_2(\text{OH})_6$ ). Insoluble in water, soluble in acids when recently precipitated, soluble in fixed alkalis, slightly soluble in  $\text{NH}_4\text{OH}$ , insoluble in  $\text{NH}_4\text{Cl}$ .

Aluminum oxide ( $\text{Al}_2\text{O}_3$ ). Ignited practically insoluble in acids.

Aluminum potassium sulphate ( $\text{KAl}(\text{SO}_4)_2, 12\text{H}_2\text{O}$ ) (alum.) Soluble in 6 parts of water.

Ammonium chloride ( $\text{NH}_4\text{Cl}$ ). Soluble in 3 parts cold, 1 part hot, water.

Ammonium carbonate (commercial). Soluble in 4 parts water.

Ammonium magnesium phosphate ( $\text{NH}_4\text{MgPO}_4, 6\text{H}_2\text{O}$ ). Insoluble in water containing ammonia, soluble in acids.

Ammonium nitrate ( $\text{NH}_4\text{NO}_3$ ). Soluble in  $\frac{1}{2}$  part water.

Ammonium sulphate ( $(\text{NH}_4)_2\text{SO}_4$ ). Soluble in 1.3 parts water.

Antimony (Sb). Insoluble in HCl out of contact with air, insoluble in dilute or concentrated  $\text{H}_2\text{SO}_4$ , cold, soluble in hot concentrated  $\text{H}_2\text{SO}_4$ , oxidized by nitric acid but not dissolved, soluble in aqua regia, insoluble in alkalis.

Antimony oxides ( $\text{Sb}_4\text{O}_6$ ,  $\text{Sb}_2\text{O}_4$ ,  $\text{Sb}_2\text{O}_5$ ) are insoluble in water, soluble in HCl, soluble in concentrated alkalis.

Antimony trisulphide ( $\text{Sb}_2\text{S}_3$ ). Insoluble in water and

dilute acids, soluble in concentrated HCl, easily soluble in dilute KOH, NaOH,  $(\text{NH}_4)_2\text{S}$ ,  $\text{K}_2\text{S}$ .

Arsenic (As). Insoluble in water, dilute HCl, dilute  $\text{H}_2\text{SO}_4$ , oxidized by concentrated  $\text{H}_2\text{SO}_4$ ,  $\text{HNO}_3$  and aqua regia.

Arsenic trioxide ( $\text{As}_2\text{O}_3$ ). Almost insoluble in water, easily soluble in alkali hydrates and carbonates, slightly soluble in acids.

Arsenic trisulphide ( $\text{As}_2\text{S}_3$ ). Insoluble in water, dilute acids, and concentrated HCl; decomposed by  $\text{HNO}_3$  or aqua regia, easily soluble in caustic or carbonated alkalies.

Barium carbonate ( $\text{BaCO}_3$ ). Insoluble in cold water, soluble in  $\text{H}_2\text{CO}_3$ . Aq, easily soluble in dilute acids.

Barium chloride ( $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ ). Soluble in 3 parts water, less soluble in dilute HCl.

Barium chromate ( $\text{BaCrO}_4$ ). Insoluble in water and in potassium dichromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ ) solution, soluble in  $\text{HNO}_3$ , HCl, and chromic acid ( $\text{H}_2\text{CrO}_4$ ).

Barium sulphate ( $\text{BaSO}_4$ ). Insoluble in water, very slightly soluble in  $\text{HNO}_3$ , insoluble in dilute HCl or KOH, slightly soluble in a hot acid solution of  $\text{Fe}_2\text{Cl}_6$ .

Bismuth (Bi). Not attacked by water or HCl in absence of oxygen, or by dilute  $\text{H}_2\text{SO}_4$ ; easily soluble in dilute nitric acid or aqua regia.

Bismuth trisulphide ( $\text{Bi}_2\text{S}_3$ ). Insoluble in water, soluble in dilute  $\text{HNO}_3$  and concentrated HCl, insoluble in alkalies and almost insoluble in alkaline sulphides.

Cadmium (Cd). Insoluble in  $\text{H}_2\text{O}$ , soluble in HCl and dilute  $\text{H}_2\text{SO}_4$ , easily soluble in  $\text{HNO}_3$ .

Cadmium sulphide ( $\text{CdS}$ ). Insoluble in  $\text{H}_2\text{O}$ , soluble in cold concentrated HCl, in  $\text{HNO}_3$ , and in boiling dilute  $\text{H}_2\text{SO}_4$ .

Calcium carbonate ( $\text{CaCO}_3$ ). Insoluble in water, soluble in dilute acids,  $\text{NH}_4\text{Cl}$ , and  $\text{H}_2\text{CO}_3$  aq.

Calcium chloride ( $\text{CaCl}_2$ ). Soluble in 1.5 parts of water.

Calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ). Slightly soluble in cold water, less in hot.

Calcium oxalate ( $\text{CaC}_2\text{O}_4$ ). Insoluble in water, soluble in mineral acids, soluble in  $\text{MgCl}_2$ , insoluble in  $\text{MgC}_2\text{O}_4$ .

Calcium phosphate ( $\text{Ca}_3(\text{PO}_4)_2$ ). Insoluble in water, soluble in acids.

Calcium sulphate ( $\text{CaSO}_4$ ). Soluble in 500 parts of water, more soluble in dilute  $\text{HCl}$ .

Chromic hydroxide ( $\text{Cr}_2(\text{OH})_6$ ). Insoluble in water, easily soluble in acids and cold caustic alkalis.

Cobalt (Co). Insoluble in  $\text{H}_2\text{O}$ , soluble in  $\text{HCl}$ ,  $\text{H}_2\text{SO}_4$ , and  $\text{HNO}_3$ .

Cobaltous sulphide ( $\text{CoS}$ ). Insoluble in  $\text{H}_2\text{O}$ , alkalis, alkaline carbonates, or sulphides; soluble in concentrated acids, particularly nitric acid or aqua regia.

Copper (Cu). Soluble in concentrated  $\text{H}_2\text{SO}_4$  at a high temperature, insoluble in dilute, slightly soluble in  $\text{HCl}$ , readily soluble in  $\text{HNO}_3$ .

Cupric oxide ( $\text{CuO}$ ). Insoluble in water, easily soluble in acids.

Cupric sulphate ( $\text{CuSO}_4$ ). Soluble in about 5 parts of water, crystallized  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ , soluble in 2.5 parts.

Cupric sulphide ( $\text{CuS}$ ). Insoluble in  $\text{H}_2\text{O}$ , soluble in boiling  $\text{HNO}_3$ , insoluble in dilute  $\text{HCl}$  or  $\text{HNO}_3$ , insoluble in  $\text{KOH}$  or  $\text{K}_2\text{S}$ , slightly soluble in colorless or yellow ammonium sulphide.

Ferrous and ferric salts. See Iron.

Gold (Au). Insoluble in  $\text{H}_2\text{O}$ ,  $\text{HCl}$ ,  $\text{HNO}_3$ ; easily soluble in aqua regia or any mixture evolving  $\text{Cl}$  or  $\text{Br}$ , sol-

uble in mixtures of HCl and nitrates, nitric acid and chlorides; insoluble in  $\text{H}_2\text{SO}_4$ , soluble in solutions of ferric and cupric salts, soluble when finely divided in KCN.

Iodine (I). Insoluble in water, soluble in concentrated acids and in KI solution.

Iron (Fe). Soluble in HCl, slowly dissolved by  $\text{H}_2\text{SO}_4$ ,  $\text{HNO}_3$ , and  $\text{HC}_2\text{H}_3\text{O}_2$ .

Ferric hydroxide ( $\text{Fe}_2(\text{OH})_6$ ). Insoluble in water, alkalis, or ammonia salts; soluble in acids.

Ferric oxide ( $\text{Fe}_2\text{O}_3$ ). Readily soluble in hot hydrochloric acid, not easily dissolved by other acids.

Ferric phosphate ( $\text{Fe}_2(\text{PO}_4)_2, x\text{H}_2\text{O}$ ). Very slightly soluble in  $\text{H}_2\text{O}$ , soluble in mineral acids, insoluble in cold acetic acid.

Ferrous sulphate ( $\text{FeSO}_4, 7\text{H}_2\text{O}$ ). Soluble in 3 parts  $\text{H}_2\text{O}$ .

Ferrous sulphide,  $\text{FeS}$ . Soluble in dilute acids with liberation of  $\text{H}_2\text{S}$  (except nitric).

Lead (Pb). Insoluble in pure water, soluble in impure water containing carbon dioxide, chlorides, nitrates, etc.; soluble in dilute nitric acid, slightly attacked by hydrochloric acid, insoluble in dilute sulphuric, soluble in concentrated  $\text{H}_2\text{SO}_4$ .

Lead chloride ( $\text{PbCl}_2$ ). Slightly soluble in cold water, more soluble in hot; slightly soluble in HCl, soluble in dilute  $\text{HNO}_3$  and in KOH.

Lead chromate ( $\text{PbCrO}_4$ ). Insoluble in  $\text{H}_2\text{O}$  and acetic acid, soluble in  $\text{K}_2\text{C}_2\text{O}_7$ , KOH, and NaOH; slightly soluble in nitric acid.

Lead oxide (litharge) ( $\text{PbO}$ ). Insoluble in  $\text{H}_2\text{O}$ , soluble in acids, soluble in KOH, NaOH, and  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ .

Lead oxide (red lead) ( $\text{Pb}_3\text{O}_4$ ). Insoluble in  $\text{H}_2\text{O}$ , acids give  $\text{PbO}_2$  and salts of  $\text{PbO}$ .



Lead sulphate ( $\text{PbSO}_4$ ). Insoluble in water, slightly soluble in  $\text{H}_2\text{SO}_4$ , insoluble in alcohol, slightly soluble in  $\text{HCl}$  and  $\text{HNO}_3$ .

Lead sulphide ( $\text{PbS}$ ). Insoluble in  $\text{H}_2\text{O}$ , dilute acids, alkalies, and alkaline sulphides; decomposed by concentrated  $\text{HNO}_3$  or aqua regia to  $\text{PbSO}_4$ ; soluble in hot concentrated  $\text{HCl}$ .

Magnesium ( $\text{Mg}$ ). Decomposes water slowly at  $100^\circ \text{C}$ ., soluble in dilute acids.

Magnesium chloride ( $\text{MgCl}_2$ ). Soluble in 1 part water.

Manganese dioxide ( $\text{MnO}_2$ ). Insoluble in water, nitric acid, or dilute sulphuric acid except in presence of reducing agents; readily dissolved by  $\text{HCl}$  and either sulphurous acid ( $\text{H}_2\text{SO}_3$ ) or oxalic acid ( $\text{H}_2\text{C}_2\text{O}_4$ ).

Mercury ( $\text{Hg}$ ). Not attacked by boiling concentrated  $\text{HCl}$  or dilute  $\text{H}_2\text{SO}_4$ , easily soluble in dilute or concentrated  $\text{HNO}_3$ .

Mercurous chloride ( $\text{Hg}_2\text{Cl}_2$ ). Insoluble in cold water, insoluble in cold dilute acids, slowly soluble on heating.

Mercuric sulphide ( $\text{HgS}$ ). Insoluble in  $\text{H}_2\text{O}$ , soluble in  $\text{K}_2\text{S}$ , insoluble in  $(\text{NH}_4)_2\text{S}$ , insoluble in  $\text{HCl}$  and  $\text{HNO}_3$ , soluble in aqua regia.

Nickel ( $\text{Ni}$ ). Slowly dissolved by  $\text{HCl}$  or  $\text{H}_2\text{SO}_4$ , readily soluble in  $\text{HNO}_3$ , insoluble in  $\text{NaOH}$ .

Nickel sulphide ( $\text{NiS}$ ). Insoluble in  $\text{H}_2\text{O}$  and dilute acids, soluble in aqua regia, slightly soluble in alkaline sulphides.

Phosphorus ( $\text{P}$ ). Insoluble in water, very soluble in  $\text{CS}_2$ , oxidized by hot concentrated  $\text{HNO}_3$ , decomposed by boiling with caustic alkalies.

Platinum ( $\text{Pt}$ ). Insoluble in  $\text{H}_2\text{O}$ ,  $\text{H}_2\text{SO}_4$ ,  $\text{HCl}$ ,  $\text{HNO}_3$ ; soluble in aqua regia.

Potassium carbonate ( $K_2CO_3$ ). Soluble in 1 part  $H_2O$ .

Potassium chlorate ( $KClO_3$ ). Soluble in 16 parts  $H_2O$ .

Potassium chloride ( $KCl$ ). Soluble in 3 parts  $H_2O$ .

Potassium chromate ( $K_2CrO_4$ ). Soluble in 2 parts  $H_2O$ .

Potassium dichromate ( $K_2Cr_2O_7$ ). Soluble in 10 parts of water.

Potassium cyanide ( $KCN$ ). Very soluble in water, insoluble in absolute alcohol.

Potassium ferricyanide ( $K_3Fe(CN)_6$ ). Soluble in 3 parts of water.

Potassium ferrocyanide ( $K_4FeCy_6, 3H_2O$ ), soluble in 4 parts of water, insoluble in alcohol.

Potassium hydroxide ( $KOH$ ). Soluble in 1 part  $H_2O$ .

Potassium iodide ( $KI$ ). Soluble in 7 parts  $H_2O$ .

Potassium nitrate ( $KNO_3$ ). Soluble in 5 parts  $H_2O$ .

Potassium sulphate ( $K_2SO_4$ ). Soluble in 10 parts  $H_2O$ .

Silica ( $SiO_2$ ). Insoluble in water and acids except hydrofluoric ( $HF$ ), soluble in hot caustic and carbonated alkalies.

Silver ( $Ag$ ). Insoluble in  $H_2O$ ,  $HCl$ , and  $HC_2H_3O_2$ ; readily soluble in  $HNO_3$  and in hot concentrated  $H_2SO_4$ ; slightly soluble in dilute  $H_2SO_4$ .

Silver arsenate ( $Ag_3AsO_4$ ). Insoluble in water, soluble in acids and ammonia.

Silver bromide ( $AgBr$ ). Insoluble in water or nitric acid, slightly soluble in concentrated sulphuric acid, soluble in  $KCN$  and strong ammonia.

Silver chloride ( $AgCl$ ). Nearly insoluble in water,  $HCl$ ,  $HNO_3$ ; soluble in  $NH_4OH$ ,  $KCN$ , and  $Na_2S_2O_3$ ; slightly soluble in  $Hg(C_2H_3O_2)_2$  and solutions of soluble chlorides.

Silver sulphide ( $Ag_2S$ ). Insoluble in  $H_2O$ , soluble in concentrated  $HNO_3$ , insoluble in  $NH_4OH$ .

Silver sulphocyanide ( $AgSCN$ ). Insoluble in water,

$\text{AgNO}_3$ , ammonium sulphocyanide ( $\text{NH}_4\text{SCN}$ ), or acids except concentrated  $\text{H}_2\text{SO}_4$  or  $\text{HNO}_3$ ; soluble in concentrated  $\text{NH}_4\text{OH}$  and  $\text{Hg}(\text{NO}_3)_2$ .

Sodium bicarbonate ( $\text{NaHCO}_3$ ). Soluble in 10 parts  $\text{H}_2\text{O}$ .

Sodium carbonate ( $\text{Na}_2\text{CO}_3$ ). Soluble in 4 parts  $\text{H}_2\text{O}$ .

Sodium chloride ( $\text{NaCl}$ ). Ten parts of water at  $15^\circ \text{C}$ . dissolves 35.9 parts.

Sodium hydroxide ( $\text{NaOH}$ ). Soluble in  $\frac{1}{2}$  part  $\text{H}_2\text{O}$ .

Sodium nitrate ( $\text{NaNO}_3$ ). Soluble in 2 parts  $\text{H}_2\text{O}$ .

Sodium sulphate ( $\text{Na}_2\text{SO}_4$ ). Soluble in 7 parts  $\text{H}_2\text{O}$ .

Sodium thiosulphate (hypo) ( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ). Soluble in 1.5 parts of water.

Sulphur (S). Insoluble in water, soluble in CS, oil of turpentine, and wood-spirit.

Tin (Sn). Slowly soluble in cold dilute  $\text{HCl}$ , easily soluble if concentrated and hot, slowly soluble in hot dilute  $\text{H}_2\text{SO}_4$ , converted to insoluble hydrated  $\text{SnO}_2$  by nitric acid, insoluble in water, soluble in alkalies.

Stannic acid ( $\text{H}_2\text{SnO}_3$ ). Insoluble in water, soluble in  $\text{HCl}$ ,  $\text{H}_2\text{SO}_4$ , and  $\text{KOH}$ ; insoluble in very dilute nitric acid.

Stannous chloride ( $\text{SnCl}_2$ ). Soluble in water and dilute hydrochloric acid.

Titanic acid ( $\text{TiO}_2 \cdot x \text{H}_2\text{O}$ ). Insoluble in water, soluble in cold  $\text{HCl}$  or  $\text{H}_2\text{SO}_4$ , precipitated on boiling dilute solution.

Zinc (Zn). Soluble in all acids, easily soluble in alkalies, and soluble in many metallic salts with precipitation of the metal.

Zinc oxide ( $\text{ZnO}$ ). Insoluble in water, soluble in acids, when moist soluble in alkalies.

Zinc sulphide ( $\text{ZnS}$ ). Insoluble in  $\text{H}_2\text{O}$ , alkali hydrates, carbonates, and sulphides; soluble in dilute  $\text{HCl}$  and  $\text{HNO}_3$ , practically insoluble in acetic acid.

Zinc sulphate ( $\text{ZnSO}_4$ ). Soluble in 2 parts of water.

## TABLE OF FACTORS.\*

ATOMIC WEIGHTS BASED ON  $O = 16$ , TAKEN FROM THE J. AM. CHEM. SOC. FOR MARCH, 1896 (F. W. CLARKE).

|   | REQUIRED.                      | FACTOR.  | LOGARITHM. |
|---|--------------------------------|----------|------------|
| AlPO <sub>4</sub>   | Al                             | .221976  | 1.8468071  |
|   | Al <sub>2</sub> O <sub>3</sub> | .418489  | 1.6216835  |
| Sb <sub>2</sub> O <sub>3</sub>                            | Sb                             | .790067  | 1.8976643  |
| Sb <sub>2</sub> S <sub>3</sub>                            | Sb                             | .714570  | 1.8540446  |
| As <sub>2</sub> S <sub>3</sub>                            | As                             | .609522  | 1.7849890  |
| Mg <sub>2</sub> As <sub>2</sub> O <sub>7</sub>            | As                             | .483268  | 1.6841870  |
| Ag <sub>2</sub> AsO <sub>4</sub>                          | As                             | .162234  | 1.2101418  |
| BaSO <sub>4</sub>   | BaO                            | .657088  | 1.8176234  |
|   | SO <sub>3</sub>                | .342912  | 1.5351829  |
|   | S                              | .187842  | 1.1878121  |
| Bi <sub>2</sub> O <sub>3</sub>                            | Bi                             | .896600  | 1.9525990  |
| CaCO <sub>3</sub>   | CaO                            | .560296  | 1.7484173  |
| CaSO <sub>4</sub>   | CaO                            | .411899  | 1.6147904  |
|   | CaCO <sub>3</sub>              | .735145  | 1.8663731  |
| CO <sub>2</sub>   | C                              | .272893  | 1.4359916  |
| Cr <sub>2</sub> O <sub>3</sub>                            | Cr                             | .684791  | 1.8355581  |
| 3K <sub>2</sub> SO <sub>4</sub> , 2CoSO <sub>4</sub>      | Co                             | .141511  | 1.1507892  |
| CuO   | Cu                             | .798995  | 1.9025440  |
| Cu <sub>2</sub> S   | Cu                             | .798644  | 1.9023531  |
| Fe <sub>2</sub> O <sub>3</sub>                            | Fe                             | .700076  | 1.8451446  |
| Fe  | Fe <sub>2</sub> O <sub>3</sub> | 1.428420 | 0.1548554  |
|   | FeO                            | 1.285610 | 0.1091100  |
|   | Fe <sub>3</sub> O <sub>4</sub> | 1.380820 | 0.1401859  |
| PbCrO <sub>4</sub>  | Pb                             | .640500  | 1.8065193  |
| PbSO <sub>4</sub>   | Pb                             | .682927  | 1.8343742  |
| Mg <sub>3</sub> P <sub>2</sub> O <sub>7</sub>             | P                              | .278681  | 1.4451076  |
|   | P <sub>2</sub> O <sub>5</sub>  | .638038  | 1.8048465  |
|   | MgO                            | .361962  | 1.5586631  |
|   | MgCO <sub>3</sub>              | .757343  | 1.8792934  |
| Mn <sub>2</sub> O <sub>3</sub>                            | Mn                             | .720490  | 1.8576283  |
| Mn <sub>2</sub> P <sub>2</sub> O <sub>7</sub>             | Mn                             | .887226  | 1.6879648  |
| (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub>         | Pt                             | .429205  | 1.6426669  |
|   | N                              | .063281  | 2.8012744  |
|   | NH <sub>3</sub>                | .076911  | 2.8859881  |
|   | NH <sub>4</sub> Cl             | .241235  | 1.3824396  |
| Pt from (NH <sub>4</sub> ) <sub>2</sub> PtCl <sub>6</sub> | N                              | .144081  | 1.1586075  |
|   | NH <sub>3</sub>                | .175114  | 1.2433212  |
|   | NH <sub>4</sub> Cl             | .549253  | 1.7397727  |
| K <sub>2</sub> PtCl <sub>6</sub>                          | KCl                            | .306951  | 1.4870695  |
|   | K <sub>2</sub> O               | .193944  | 1.2876767  |
| KCl   | K <sub>2</sub> O               | .631840  | 1.8006072  |
| K <sub>2</sub> SO <sub>4</sub>                            | K <sub>2</sub> O               | .540593  | 1.7328706  |
| SiO <sub>2</sub>  | Si                             | .470199  | 1.6722814  |

\* J. Am. Chem. Soc., Oct., 1896.

276 TABLE OF VALUES OF N/100 PERMANGANATE.

|   | REQUIRED.         | FACTOR. | LOGARITHM. |
|---|-------------------|---------|------------|
| AgBr  | Br                | .425560 | 1.6289611  |
| AgI   | I                 | .540813 | 1.7326479  |
| AgCl  | Cl                | .247262 | 1.3931579  |
|   | Ag                | .752788 | 1.8766436  |
| NaCl  | Na <sub>2</sub> O | .530769 | 1.7249057  |
| Na <sub>2</sub> SO <sub>4</sub>               | Na <sub>2</sub> O | .436801 | 1.6402836  |
| SnO <sub>2</sub>                              | Sn                | .788150 | 1.8966087  |
| TiO <sub>2</sub>                              | Ti                | .600749 | 1.7786928  |
| ZnO   | Zn                | .803464 | 1.9049663  |
| Zn <sub>2</sub> P <sub>2</sub> O <sub>7</sub> | Zn                | .429115 | 1.6325737  |
| ZnNH <sub>4</sub> PO <sub>4</sub>             | Zn                | .366438 | 1.5640011  |

TABLE OF VALUES OF N/100 PERMANGANATE SOLUTION.\*

$K_2Mn_2O_8 = 3.162$  GRAMS PER LITRE.

|  | Grams per Cubic Centimeter. |
|--|-----------------------------|
| $K_2Mn_2O_8$ .....   | .003162                     |
| Fe.....  | .005602                     |
| $Fe_2O_3$ .....  | .008002                     |
| FeO.....   | .007202                     |
| $Fe_3O_4$ .....  | .007735                     |
| $FeSO_4 \cdot 7H_2O$ .....                                   | .027820                     |
| $FeSO_4(NH_4)_2SO_4 \cdot 6H_2O$ .....                       | .039240                     |
| $H_2C_2O_4 \cdot 2H_2O$ .....                                | .006303                     |
| $(NH_4)_2C_2O_4 \cdot H_2O$ .....                            | .007109                     |
| CaO from $CaC_2O_4$ .....                                    | .002804                     |
| $CaCO_3$ " ".....  | .005004                     |
| $CaSO_4 \cdot 2H_2O$ from $CaC_2O_4$ .....                   | .008609                     |
| Mn (Volhard).....  | .0016497                    |
| Mn (Ford-Williams).....                                      | .0027495                    |
| P (Noyes).....   | .000086167                  |
| $P_2O_5$ (Noyes).....  | .00019728                   |
| Sn from $SnCl_2$ .....                                       | .0059525                    |
| $H_2S$ .....   | .0017043                    |
| $H_2O_2$ .....   | .0017008                    |
| I.....   | .0012685                    |
| Equivalent to N/60 $K_2Cr_2O_7$ , 4.9083 gms. per litre..... | .004908                     |

Factor to convert Fe standard to P standard (Noyes) is 0.0153814 ; to Mn standard (Volhard) is 0.294484.

\* Atomic masses according to J. Am. Chem. Soc., March, 1896, 0 = 16.

# EXAMPLES AND PROBLEMS.

1. Calculate assay ton for a long ton.

1 lb. avoirdupois = 16 oz. of 437.5 grams each = 7000 grains ; 1 long ton = 2240 lbs.

1 oz. troy = 480 grains.

$$\frac{2240 \times 7000}{480} = 32,666 \text{ ounces troy in a long ton.}$$

$\therefore$  32.666 grams is the assay ton for a long ton.

2. How much nitric acid 1.42 sp. gr. and how much water should be mixed to give 6 litres of nitric acid 1.16 sp. gr. ?

Let  $x$  be litres of nitric acid,  $6 - x$  litres of water. 1.42  $x$  weight nitric acid + 1 ( $6 - x$ ) weight of water = 6 (1.16) weight of mixture.  $x = 2.286$  ( $6 - x$ ) = 3.714.

3. Five hundred grams of a low-grade tin ore were panned down ; the concentrates after drying weighed 24 grams. 2 portions of 10 grams each were mixed with 10 grams CuO and KCy, and fused. The buttons weighed 12.15 and 12.30 grams. A blank assay on the oxide of copper gave 7.4 grams. What is the percentage of tin ?

Average weight of buttons is 12.235 grams.

Weight of tin = 12.235 - 7.4 = 4.835 “

Total tin is  $\frac{4.835 \times 24}{10}$  or 11.604 grams = 2.32% tin.

4. An iron ore contains silica 32%, alumina 6%, lime 10%. Calculate the minimum charge for Percy's slag.

| 10 Grams of Ore.                            | Required. | Excess. |
|---|-----------|---------|
| SiO <sub>2</sub> 3.2 gms.....               | 2.5       | 0.7     |
| Al <sub>2</sub> O <sub>3</sub> 0.6 gm. .... | 1         |         |
| CaO 1.0 gm. ....                            | 3         |         |

0.8 gram of kaolin gives the required amount of alumina. The excess of silica is now  $0.7 + 0.4 = 1.1$ . Make up charge for this excess.

$$\frac{x}{2} + 1.1 : \frac{x}{2} :: 5 : 2; \quad x = 1.466.$$

$$\text{Lime to add is } 2 + \frac{3}{2} 1.466 = 4.2.$$

$$\text{Kaolin to add } 0.8 + 1.466 = 2.266.$$

$$\text{Proof: SiO}_2 \quad 3.2 + 0.4 + 0.733 = 4.333 \div 1.733 = 2.5.$$

$$\text{Al}_2\text{O}_3 \quad 0.6 + 0.4 + 0.733 = 1.733 \div 1.733 = 1.$$

$$\text{CaO} \quad 1.0 + 4.2 = 5.2 \div 1.733 = 3.$$

5. An ore of nickel, cobalt, and copper gave the following results from 5 gms. of the original sample :

a. Weight of arsenides from fusion . . . . 2.467 gms.

b.    "    " arsenide of cobalt, nickel,  
                    and copper . . . . . 1.246   "

c.    "    " arsenide of nickel and  
                    copper . . . . . 0.542   "

d.    "    " button of copper and gold 0.421   "

e.    "    " gold added . . . . . 0.150   "

Calculate the per cent. of nickel, cobalt, and copper in the original ore.

$$d - e = \text{weight of Cu } 0.271; \quad 0.271 \text{ Cu} = 0.3746 \text{ Cu}_3\text{As}_2.$$

$$c - 0.3746 = 0.1674 \text{ weight of Ni}_3\text{As}_2 = 0.1020 \text{ gm. Ni.}$$

$$b - c = 0.704 \text{ weight of Co}_3\text{As}_2 = 0.430 \text{ gm. Co.}$$

$$\text{Cu } 0.271 \div 5 \text{ and multiplied by } 100 = 5.42\% \text{ copper.}$$

$$\text{Ni } 0.099 \text{ " " " " " } 2.04\% \text{ nickel.}$$

$$\text{Co } 0.433 \text{ " " " " " } 8.60\% \text{ cobalt.}$$

6. A sample submitted for assay gave, on being pulverized and passed through a sieve of 80 meshes to the linear inch, the following weights :

|                          |              |
|--------------------------|--------------|
| A. Weight of sample..... | 1499.07 gms. |
| B. Scales of metal.....  | 40.75 "      |
| C. Sifted ore.....       | 1458.32 gms. |

It being known from the mineralogical composition of the sample that it was a rich ore,  $\frac{1}{3}$  A.T. was taken for an assay of the sifted portion (C). The residue of metallic scales, etc. (B), was scorified with test-lead, and yielded a button weighing 60.35 gms. This button was rolled out, and two average samples of 10 gms. each were cupelled.

The following results were obtained from the complete assays :

C. SIFTED ORE—CRUCIBLE ASSAY.

One third assay ton, 9.722 gms., yielded :

|                      | 1       | 2       | Average. |
|----------------------|---------|---------|----------|
| Au + Ag.....         | 0.19355 | 0.19275 | 0.19315  |
| Au (by parting)..... | 0.00025 | 0.00025 | 0.00025  |
| Ag.....              | 0.19330 | 0.19250 | 0.19280  |
| Ag in litharge.....  | 0.00067 | 0.00067 | 0.00067  |
| Ag in ore.....       | 0.19263 | 0.19183 | 0.19213  |

The litharge yielded one milligram silver per assay ton, and two thirds assay ton were employed.

B. METALLIC SCALES.

10 grams of the scorified button yielded :

|                      | 1      | 2      | Average. |
|----------------------|--------|--------|----------|
| Au + Ag.....         | 5.0625 | 5.0620 | 5.0622   |
| Au (by parting)..... | 0.0020 | 0.0020 | 0.0020   |
| Ag.....              | 5.0605 | 5.0600 | 5.0602   |
| Ag in test-lead..... | none   | none   | none     |

CALCULATION:

$$\text{C. Sifted ore} \dots 1458.32 \times \frac{0.19213}{9.722} = 28.819 \text{ Ag.}$$



$$\text{B. Metallic scales } 40.75, \quad \frac{5.0602}{10} \times 60.35 = 30.538 \text{ Ag.}$$

$$\text{A. Total ore } \dots \overline{1499.07} \qquad \qquad \qquad \overline{59.357} \text{ Total Ag.}$$

$$29166.66 \times \frac{59.357}{1499.07} = 1154.71 \text{ oz. per 2000 lbs.}$$

$$\text{C. Sifted ore } \dots \overline{1458.32} \times \frac{0.00025}{9.722} = 0.0375 \text{ Au.}$$

$$\text{B. Metallic scales } \dots 40.75, \quad \frac{0.002}{10} \times 60.35 = 0.0121 \text{ Au.}$$

$$\text{A. Total ore } \dots \overline{1499.07} \qquad \qquad \qquad \overline{0.0496} \text{ Total Au.}$$

$$29166.66 \times \frac{0.0496}{1499.07} = 0.97 \text{ oz. per 2000 lbs.}$$

*Result per 2000 lbs. Ore.*

Silver.....  $\overline{1154.71}$  oz. at \$0.65 ..... \$750.56

Gold..... 0.97 oz. at \$20.67 .....  $\overline{20.04}$

Total bullion.....  $\overline{1155.97}$  oz.  $\overline{\$770.60}$

7. A preliminary assay on 5 grams of ore gave lead reduced 14 grams. How much nitre (oxidizing power 4) should be added to give a button of 18 grams when 1 A.T. of ore is used.

1 A.T. would reduce  $6 \times 14 \text{ grams} = 84$ ;  $84 - 18 = 66$  grams of lead to be oxidized;  $66 \div 4 = 16.5$  grams of nitre.

8. An ore gave no button from preliminary assay on 5 grams, but was known to contain manganese; 2 grams of argol (R. P. 10) were then added to the charge and the assay repeated; the button weighed 16 grams. What amount of argol should be added to 2 A.T. of ore? 5 grams of ore oxidizes 4 grams of lead; 2 A.T. will oxidize 48; we must add, therefore, enough argol to offset this and to reduce 18 grams of lead  $(48 + 18) \div 10 = 6.6$  grams argol.

9. How much water should be added to 6 litres of a

salt solution to make it normal when 98.6 c.c. of the solution precipitates 1 gram of pure silver?

$$6000 : x :: 98.6 : 1.4 ; x = 85.19 \text{ c.c.}$$

10. How much salt should be added to 6 litres of a salt solution to make it normal when 100.6 c.c. precipitates 1 gram pure silver?

$$6000 : x : 100.6 : 6 ; x = 35.78 \text{ c.c., the water in excess.}$$

$$100 : 35.78 :: .54207 : x.$$

$x = 0.19385$ , the salt in grams to be added for the excess of water.

11. An alloy cupelled and parted gave 0.9848 gram of silver in 1 gram after correction. Added in volumetric test:

|                           |          |
|---------------------------|----------|
| Normal salt solution..... | 100 c.c. |
| Decime " " .....          | 5 "      |
| " silver " .....          | 2 "      |

Decime solutions added 1 c.c. at a time and in the order stated.

Strength of normal salt solution 99.7 c.c. = 1 gram silver.

What was the amount taken for assay and the fineness of the alloy?

$$\frac{1000}{0.9848} = 1.0154, \text{ weight taken for volumetric assay.}$$

Salt used was 100.4 c.c.

$$99.7 : 1 :: 100.4 : x ; x = \text{amount of silver} = 1.007 \text{ grams.}$$

$$1.0154 : 1.007 :: 100 : y ; y = \text{fineness} = 991.7.$$

12. 1 gram of an alloy cupelled, parted, and corrected gave silver 984.2 mgs., gold 8.4 mgs. Wet assay: added normal salt 100 c.c., decime salt 13 c.c., decime silver 3 c.c.

Strength of normal salt 101.2 c.c. = 1 gram silver.

Calculate the fineness of the bullion.

Salt actually used 101.1 c.c.

$$101.2 : 1 :: 101.1 : x ; x = \text{silver found} = 0.999 \text{ grams.}$$

$984.2 : 1000 :: 1 : x$ , weight taken for assay = 1.016 grams.

$1.016 : 0.999 : 1000 : x$ ;  $x = 983.27$  fineness.

13. 500 milligrams of gold bullion cupelled and parted gave a cornet weighing 458.35 milligrams. The surcharge was + 0.3. The fineness before correction be will 916.7.

Then  $x$ , the fineness, will be  $\frac{916.7 \times 1000}{1000 + 0.3}$  or 916.424.

14. 1 gram of a platinum alloy was treated according to the "First method"; after treating with nitric acid the residue weighed 0.15073 grams, after treating with dilute aqua regia 0.0102 grams, after treating with strong aqua regia 0.0055 grams. The precipitated gold weighed 0.03508 grams. Calculate the per cent. of Pt, Au, Ir, and iridosmium.

The final residue is iridosmium  $0.0055 \times 100 = 0.55\%$ .

The gold is  $0.03508 \times 100$  or 3.508%.

$0.0102 - 0.0055$  or 0.0047 is the iridium = 0.47%.

$0.15073 - (0.03508 + 0.0102)$  is the platinum = 0.1055 or 10.55%.

15. 1 gram of a platinum alloy treated by the "Second method" gave as follows:

|   |         |       |
|---|---------|-------|
| Silver added first cupellation.....   | 0.4724  | grams |
| Weight of button.....   | 0.9491  | "     |
| Weight of residue after parting with $H_2SO_4$ .                                  | 0.15142 | "     |
| Weight of residue after alloying with silver<br>and parting with nitric acid..... | 0.05116 | "     |
| Weight of final residue.....  | 0.00234 | "     |

The silver is  $0.9491 - (0.4724 + 0.15142)$  or 0.3253 grams = 32.53%.

The platinum is  $0.15142 - 0.05116$  or 0.10026 = 10.026%.

The gold is  $0.05116 - 0.00234 = 4.88\%$ .

Iridosmium 0.234%.

16. 1 gram of zinc ore gave 0.3417 grams of  $\text{Zn}_2\text{P}_2\text{O}_7$ .  
What is the percentage of zinc?

$$\text{Zn}_2\text{P}_2\text{O}_7 (304.86) : 2\text{Zn} (130.82) :: 0.3417 : x.$$

$$x = 0.1466 \text{ or } 14.66\% \text{ zinc.}$$

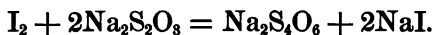
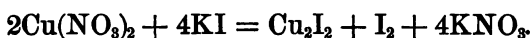
17. What is the factor to convert  $\text{SnO}_2$  to tin?

$$\text{Sn} = 119, \text{O} = 16. \therefore \text{SnO}_2 \text{ is } \frac{119}{158} \text{ tin, or wt. SnO}_2 \times 0.788 = \text{wt. Sn.}$$

18.  $\frac{1}{2}$  gram of a copper matte required 28.2 c.c. of a cyanide solution, whose strength was 1 c.c. = 0.0104 gms. Cu. What is the percentage of copper?

$$\text{Copper found} = 28.2 \times 0.0104 = 0.2933 = 58.66\%.$$

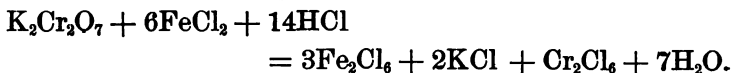
19. How much crystallized sodium thiosulphate ( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ), must be dissolved in water and diluted to one litre to give a solution whose strength is 1 c.c. = 0.010 gms. Cu?



1 atom of copper corresponds to 1 molecule of thiosulphate.

$$63.6 : 248 :: 10 : x. \quad \text{Ans. 39 grams.}$$

20. What is the strength in terms of iron of a solution of  $\text{K}_2\text{Cr}_2\text{O}_7$  containing 5 grams to the litre?



Hence 1 molecule of  $\text{K}_2\text{Cr}_2\text{O}_7$  oxidizes 6 atoms of ferrous iron to the ferric condition.

$$294.5 : 336.12 :: 0.005 : x.$$

$$x = \text{Fe per c.c.} = 0.005701.$$

21. (a) What is a N/50 solution of  $\text{K}_2\text{Mn}_2\text{O}_8$ ?

Such a solution will contain  $\frac{1}{50}$  of the molecular weight of permanganate in grams to the litre, or 6.32 grams.

(b) What is its strength in terms of  $\text{Fe}_2\text{O}_3$ ?

One molecule  $\text{K}_2\text{Mn}_2\text{O}_8$  corresponds to  $10\text{Fe}$ , or  $5\text{Fe}_2\text{O}_3$ .

$316 : 800 :: 0.00632 : x$ .

$x = 0.0160$  gms.  $\text{Fe}_2\text{O}_3$  per c.c.

(c) What amount of iron ore must be weighed out so that each c.c. used will read 1% of iron (Fe)?

The strength of this solution is 1 c.c. = 0.0112 gms. Fe.

Hence if 100 times this amount or 1.12 grams is used, the burette reads per cent. direct.

(d) What is the strength of this solution against manganese by Volhard's method?

One molecule  $\text{K}_2\text{Mn}_2\text{O}_8 = 10\text{Fe}$ .

One molecule  $\text{K}_2\text{Mn}_2\text{O}_8 = 3\text{MnO}_2 = 3\text{Mn}$ .

$10\text{Fe} = 3\text{Mn}$ , or Mn standard is  $\frac{3}{10}$  of Fe standard or 0.0033.

22. 1 gram of an ore containing arsenic was treated according to Pearce's method and the silver in the silver arsenate determined by titrating with  $\text{NH}_4\text{CNS}$ . The amount used was 27.9 c.c., the strength of solution 1 c.c. = 0.007 gms. Ag. What was the percentage of arsenic?

Silver was  $27.9 \times 0.007$  grams = 0.1953. From the formula of the precipitate  $\text{Ag}_3\text{AsO}_4 \cdot 3\text{Ag}$  or 324 corresponds to 1As 75, or Ag : As :: 108 : 25.  $0.1953 : x :: 108 : 25$ .  $x =$  wt. As = 0.0452 or 4.52%.

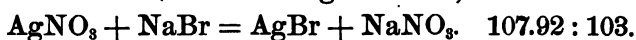
23. What is the strength against phosphorus of a N/100 solution of  $\text{K}_2\text{Mn}_2\text{O}_8$  according to Noyes' method?

$3\text{K}_2\text{Mn}_2\text{O}_8 = 5\text{Mo}_2\text{O}_3 = 30\text{Fe} = 10\text{MoO}_3$   $\therefore$  Fe standard :  $\text{MoO}_3$  standard :: 1680 : 1440 or as 7 : 6.

As the composition of the "yellow precipitate" is  $12\text{MoO}_3(\text{NH}_4)_3\text{PO}_4$ , the ratio is  $12\text{MoO}_3$  to 1P or 1728 to 31.

The iron standard of the N/100 solution is 0.0056.  $\therefore$  the P standard is  $0.0056 \left( \frac{6}{7} \times \frac{31}{1728} \right)$  or times 0.01538 = 0.000086.

24. If sodium bromide were used instead of sodium chloride in the silver bullion assay, how many grams should 1 litre contain to give a solution of the same strength as the normal salt? (100 c.c. = 1 gm. silver.)



$\therefore 10 : x :: 107.92 : 103.$  Ans. 9.544 grams.

25. 1.5 grams of coal gave by Eschka's method 0.2894 grams of  $\text{BaSO}_4$ . The oxide of magnesia used contained 0.32% S. What is the percentage of sulphur in the coal?

$0.2894 \times 0.13745$  (the factor for S in  $\text{BaSO}_4$ ) = 0.03978.  
The S in 1 gram  $\text{MgO}$  = 0.0032, S in 1.5 grams coal =  $0.03658 \div 1.5$  and multiplied by 100 = 2.44% S in coal.

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## COST OF OUTFIT FOR ORDINARY FIRE ASSAYS.

|  |                     |
|--|---------------------|
| 1. Ore-balance.....                          | \$15.00 to \$ 20.00 |
| 2. Bullion-balance.....                      | 65.00 to 110.00     |
| 3. Weights, assay ton and gram.....          | 16.00               |
| 4. Muffles, 6 at 0.75 to 1.10 each.....      | 4.50 to 6.60        |
| 5. Hessian crucibles, 50 nests small fives.. | 2.50                |
| 6. Scorifiers, 500 at 2c. each .....         | 10.00               |
| 7. Cupel-mould .....                         | 2.50                |
| 8. Scorification-moulds.....                 | .95                 |
| 9. Crucible-scorification-, and cupel-tongs  | 3.00 to 4.00        |
| 10. Hammers.....                             | 1.00 to 1.50        |
| 11. Pokers and scrapers.....                 | 1.00                |
| 12. Cutting-shears, vise, and anvil. ....    | 5.00                |
| 13. Files, chisels, saw, hatchet, etc.....   | 2.50                |
| 14. Iron mortars, large and small.....       | 3.25                |
| 15. Plate and rubber.....                    | 12.00               |
| 16. Sieves, 40, 60, 80, and 100 mesh .....   | 5.00                |
| 17. Tin sampler .....                        | .75                 |
| 18. Mixing-scoop.....                        | .35                 |

|   |                    |
|---|--------------------|
| 19. Reagent-bottles, glass-stoppered, set of 40.....      | 6.00               |
| 20. Parting-bottles.....                                  | .90                |
| 21. Bottles for samples, corked, 2 doz....                | 2.00               |
| 22. Ring-stand and lamp.....                              | 1.20               |
| 23. Wash-bottles, 1 large, 1 small.....                   | .90                |
| 24. Horn spatula, spoon, etc.....                         | .75                |
| 25. Parting-flasks, annealing-cups, etc....               | 1.50               |
| 26. Porcelain mortar.....                                 | 1.00               |
| 27. Glass rods, tubes, funnels, beakers....               | 2.50               |
| 28. Notebook, towels, brushes, etc.....                   | 1.50               |
| 29. Iron concentrating-pan, pipettes, etc..               | 4.00               |
| 30. Blowpipe, lamp, etc.....                              | 6.50               |
| 31. Hammer and anvil, small.....                          | 1.30               |
| 32. Button-brush.....                                     | .50                |
| 33. Scissors, knife, magnifier, and magnet.               | 2.20               |
| 34. Forceps and tongs.....                                | 1.75               |
| 35. Test-tubes and stand.....                             | 1.00               |
| 36. Filter-papers, test-papers, etc.....                  | 1.00               |
| 37. Platinum wire and foil.....                           | 2.00               |
| 38. Set of heavy scales with weights.....                 | 8.00               |
| 39. Bone-ash for cupels.....                              | 4.00               |
| 40. Litharge.....   | 1.00               |
| 41. Soda, borax, etc.....                                 | 1.50               |
| 42. Test-lead.....  | 5.00               |
| 43. Nitre, argol, potassium cyanide.....                  | 4.00               |
| 44. Nitric, hydrochloric, and sulphuric acids.....        | 6.00               |
| 45. Extra reagents.....                                   | 6.00               |
| 46. Furnace, portable..                                   | 30.00 to 75.00     |
| 47. Weights for assay balance, 1 gram to 1 milligram..... | 10.60              |
|   | <hr/>              |
|   | \$266.90 to 363.50 |



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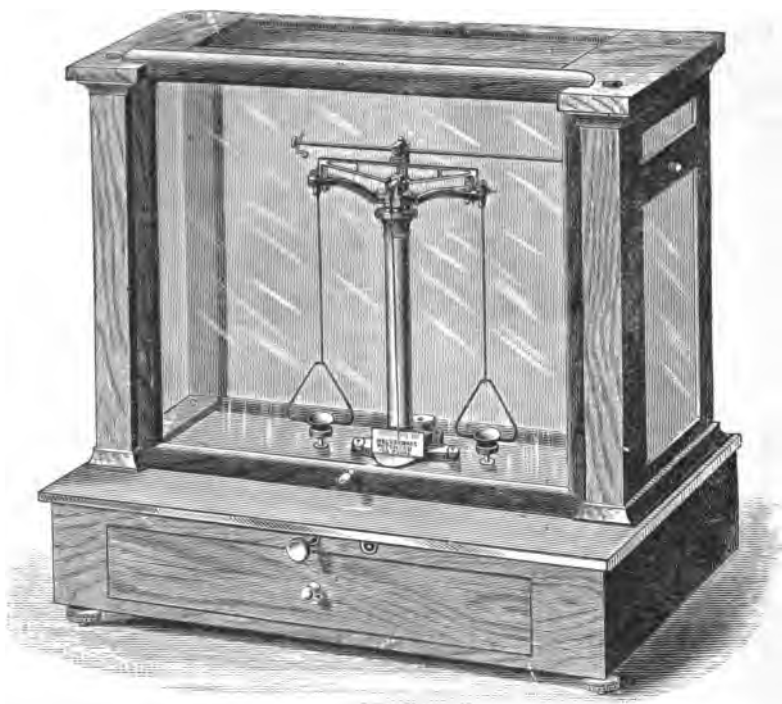




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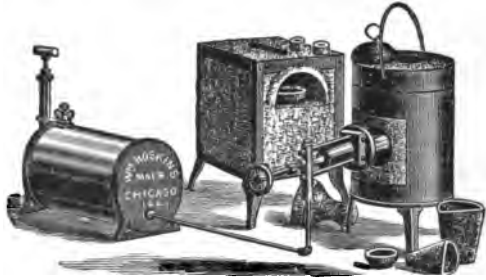
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
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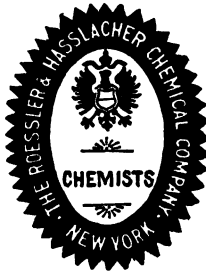
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